

Canadian Journal of Research

Issued by THE NATIONAL RESEARCH COUNCIL OF CANADA

VOL. 23, SEC. F.

NOVEMBER, 1945

NUMBER 6

DRIED MILK POWDER

II. FACTORS AFFECTING THE SORPTION OF CARBON DIOXIDE¹

BY JESSE A. PEARCE²

Abstract

Sorption of carbon dioxide by milk powder in a closed system at 35° C. and at approximately 74 cm. of mercury was observed to be greater than 0.4 cc. per gm. after 150 hr., while only 0.012 cc. of nitrogen was absorbed per gm. after 70 hr. The initial sorption of carbon dioxide varied with time according to the equation:

$$s^m = kt$$

where s is 100 times the amount sorbed in cc. per gm. at any time, t (min.), and k and m are constants peculiar to the system under investigation. The logarithmic form of this equation was used. Powders with 26, 28, and 30% fat did not differ in behaviour, but sorption curves for powders with only 1% fat had lower $\frac{\log k}{m}$ values and lower $\frac{1}{m}$ values than the curves for the high fat levels.

Powders with 1% fat sorbed carbon dioxide in an identical manner when exposed to either 100% carbon dioxide or a mixture of 20% carbon dioxide and 80% nitrogen. For whole milk powder, dilution to 80% nitrogen content was effective in reducing the initial sorption rate of carbon dioxide. Great variation was observed in the sorption behaviour of powders from different plants and in powders produced at different time intervals in the same plant. Temperature differences within the range 25° to 40° C. had no effect on sorption. Palatability and $\frac{1}{m}$ correlated to the extent of $r = .61$.

Introduction

Packing milk powder in an atmosphere of carbon dioxide or in mixed nitrogen and carbon dioxide has become common commercial practice. The use of nitrogen has been studied, but has been reported to be unfavourable (7). Carbon dioxide is believed to react with the fat (2) and its use appears to add little to the storage life of milk powder (2, 7, 8). In spite of this, carbon dioxide is still being used (3). During the development of a cellulose base container for gas packing (8), data on the sorption of carbon dioxide by milk powder were required, but little could be found in the literature. Therefore, a study of this problem was believed desirable, particularly if milk powder were to be packed in such a flexible container. The present paper records the effect of gas composition (carbon dioxide and carbon-dioxide-nitrogen mixtures), temperature, moisture content, fat content, and variations in processing on the sorption of carbon dioxide by milk powder.

¹ Manuscript received June 13, 1945.

Contribution from the Division of Applied Biology, National Research Laboratories, Ottawa. Issued as Paper No. 140 of the Canadian Committee on Food Preservation and as N.R.C. No. 1323.

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Materials

Currently available, spray dried milk powders from Canadian plants were used. Since the product deteriorates rapidly, fresh materials were obtained for each phase of the study. The moisture and fat contents are noted in Fig. 3. These powders were used without further treatment, except that, in the study of moisture effects, moisture contents were adjusted to levels between 1.8 and 5.0%.

Sorption Method

Preliminary measurements on two sample tins of commercially available milk powder showed that there was 0.57 gm. of milk solids to every cubic centimetre of container space. However, the solids occupied only 0.26 cc. of container space. The remainder must have been headspace and interstitial space, plus the space inside the hollow, spherical milk particles (5). The inclusion of this last item seems permissible since the packing density averaged about 0.86 gm. per cc. of powder.

The apparatus, shown diagrammatically in Fig. 1, permitted measurement of sorption rates at 0.3 gm. of milk solids per cubic centimetre of container space, as opposed to the value of approximately 0.6 gm. per cc. of commercial container space. Since the initial sorption rate is not dependent on container volume, it was believed that this apparatus would give information of value.

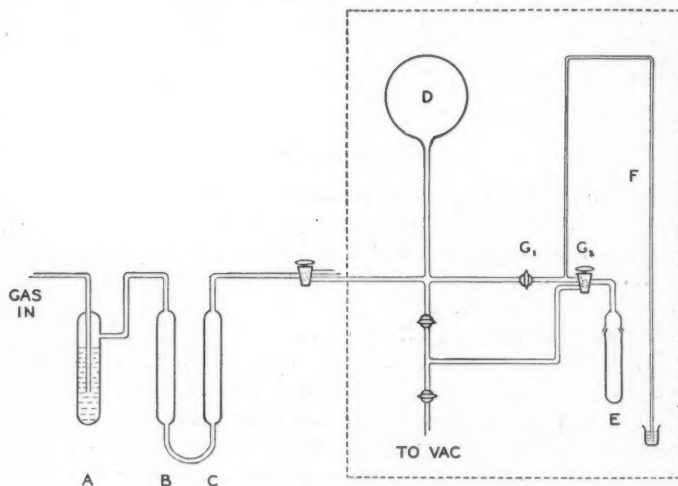


FIG. 1. Diagram of sorption apparatus.

Carbon dioxide or nitrogen was passed through the purifying train shown at A, B, and C, which represent alkaline pyrogallol (to remove oxygen), calcium chloride, and phosphorous pentoxide (both to remove water vapour). The gas or gas mixture was then stored in the five litre volume (D), which

permitted flooding of the bulb (*E*) without reducing the pressure in the sorption system by more than 1.5 cm. of mercury.

The sorption bulb (*E*) was filled with 50 gm. of milk powder and together with the manometer (*F*) evacuated through *G*₂ for 15 min. After evacuation the stopcock (*G*₁) was opened momentarily to permit pressure equalization between bulbs *D* and *E*. The pressure decreases in the system during a five-hour period were determined at logarithmic time intervals and were used to calculate the amount of gas sorbed by the milk powder.

The dotted lines indicate the portion of the apparatus enclosed in an air jacket that could be controlled at the desired temperatures (25° to 40° C.) to $\pm 1^\circ$ C. Average barometric pressure throughout the experiment was 75.7 with a range of 74.7 to 77.2 cm. of mercury. Since the expansion of gas from bulb *D* into bulb *E* reduced the pressure 1.5 cm., the average initial pressure in the system was approximately 74 cm. of mercury (Fig. 1).

In a portion of the study, palatability scores were determined by a method described previously (6). Panels of 14 tasters scored the reconstituted milk on the basis of 10 (the equivalent of whole milk) to 0 (a repulsive sample).

Results

Interpretation of Results

The curves shown in Fig. 2A depict sorption against time at 35° C. with the pressure initially at 74 cm. of mercury. Sorption is expressed as 100 times the volume of gas sorbed by a gram of milk powder. These curves indicate that carbon dioxide was extensively sorbed, while nitrogen was sorbed only to a minor extent, and that dilution of the carbon dioxide with nitrogen markedly altered the sorption curve. The amount of carbon dioxide sorbed after 150 hr. was 0.444 cc. per gm. After 70 hr. only 0.012 cc. of nitrogen was sorbed per gm. The logarithmic curve for sorption by nitrogen seems to indicate a lengthy induction period followed by an increased sorption rate. It was observed that the amount sorbed varied with time according to the equation:

$$s^m = kt,$$

where *s* is the amount sorbed per gm. at any time, *t*, in minutes and *k* and *m* are constants. This relation has been previously observed for the sorption of gases on metals, glass, etc. (1, pp. 18-20; 4). Verification of the relation for milk powder was sought by the plotting of logarithmic curves as shown in Figs. 2B, 2C, where log *s* is plotted against log *t*.

As in previous observations (1, 4) the experimental points deviated from the straight line as saturation of the sorbent was approached, or as the concentration of gas in the system decreased. As a result, in a system containing 20% carbon dioxide and 80% nitrogen, the logarithmic relation had to be determined by the sorption occurring in about the first hour. Otherwise the relation was based on the sorption curve for a five hour period.

For convenience in handling the data, values were transposed to logarithms and $\log k$ and $\frac{1}{m}$ were calculated from the equation:

$$\log s = \frac{\log k}{m} + \frac{1}{m} \log t$$

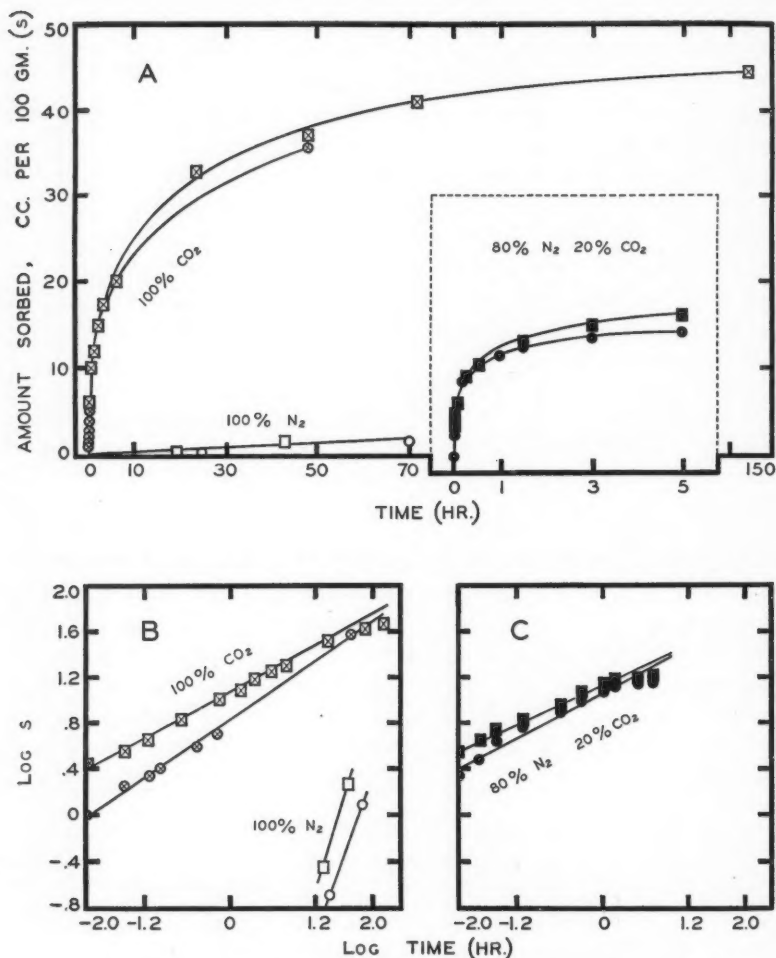


FIG. 2. Sorption of carbon dioxide and nitrogen by milk powder.

Differences between values in $\log k$ and $\frac{1}{m}$ were selected either by analyses of variance or by visual inspection. All results differing significantly were then shown as curves of $\log s$ against $\log t$.

Duplicate curves were established for a sample from each of two plants. The results, in Fig. 3A, show that by means of this apparatus it was possible to obtain good agreement between duplicates. Therefore, in subsequent work only a single curve was determined for each sample.

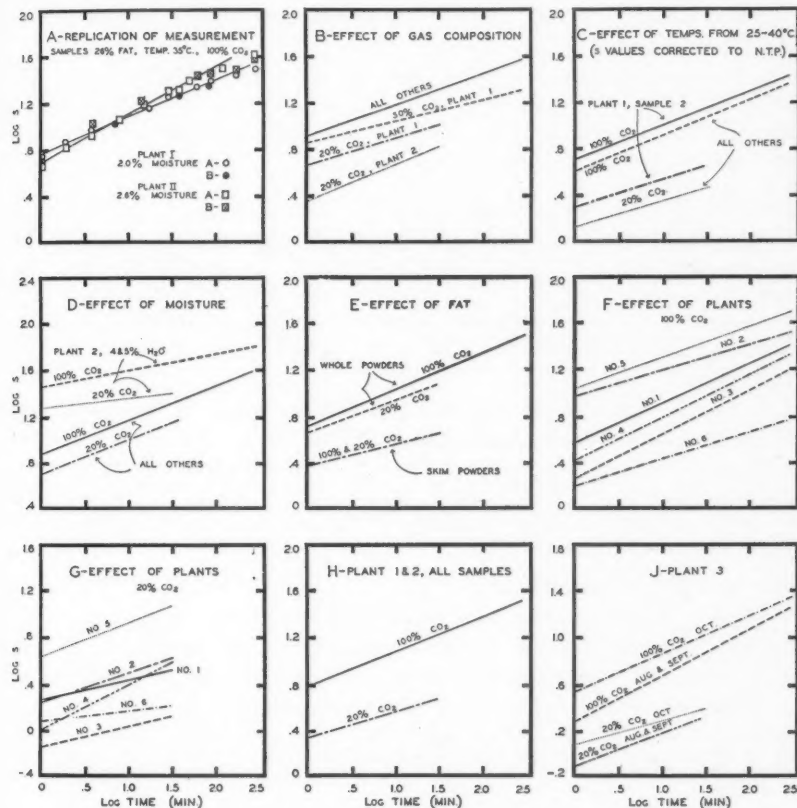


FIG. 3. Effect of various factors on the sorption of carbon dioxide by milk powder.

In general, samples of whole milk contained from about 2.0 to 2.5% moisture and 26% butterfat, while samples of skim milk contained about 3% moisture and 1% butterfat. Gases were carbon dioxide or carbon dioxide and nitrogen.

B. "All others" describes samples from Plant 1 in an atmosphere of 100 and 80% carbon dioxide, and samples from Plant 2 in an atmosphere of 100, 80, and 50% carbon dioxide.

C. "All others" describes samples from each of Plants 1 and 2.

D. "All others" describes samples of 1.8, 2.3, 3.0, 4.0, and 5.0% moisture from Plant 1, and samples of 1.8, 2.3, and 3.0% moisture from Plant 2.

E. "Whole powders" describes samples of 26 and 28% butterfat from Plant 1 and samples of 26, 28, and 30% butterfat from Plant 2.

"Skim powders" describes samples of 1% butterfat from both Plants 1 and 2.

The Effect of Temperature and Partial Pressure of Carbon Dioxide

The effect of diluting carbon dioxide with nitrogen in powders containing 26% fat from two plants is shown in Fig. 3B. Dilution with 80% nitrogen was necessary to reduce effectively the rate of sorption of carbon dioxide by milk powder. Further comparisons were made only between the effects of sorption from 100% carbon dioxide and from a mixture of 20% carbon dioxide and 80% nitrogen.

Temperature effects between 25° and 40° C. were negligible when the amount of gas sorbed was calculated to N. T. P. (Fig. 3C). Again some differences between samples were evident and differences between concentrated and diluted carbon dioxide were marked.

Effect of Moisture Content and Fat Content

Powders containing 26% fat from two plants were adjusted to moisture levels between 1.8 and 5.0%. Milk powders from one plant at 4 and 5% moisture showed alteration in the shape of the sorption curve (Fig. 3D). All other samples had normal sorption curves. It is possible that a layer of surface water was formed on these abnormal samples and dissolved some carbon dioxide.

Milk powders with fat contents of 26, 28, and 30% did not differ in either slope or constant in the logarithmic equation (Fig. 3E). However, reduction of the fat content to 1% had a significant effect and resulted in common behaviour for both samples at both carbon dioxide levels. It is apparent that carbon dioxide is sorbed not only in the butter fat, but by the other constituents of milk powder.

Differences Between and Within Plants

Further information concerning this variation observed in material produced by different plants was believed desirable. The curves in Figs. 3F and 3G show that variations in products from six plants far exceeded any of the differences observed up to this point, and show little consistency in behaviour of powders from different sources. It is also evident from Fig. 3H and 3J that considerable variation is likely to occur in products from the same source.

Relation Between Sorption and Palatability

It was of interest to evaluate the relation between constants of the logarithmic equation and the palatability of the powders. The correlations have been determined as follows: palatability and $\log k$, $r = -.26$; palatability and $\frac{\log k}{m}$, $r = .07$; palatability and $\frac{1}{m}$, $r = .61^*$. This correlation is higher than any observed previously for objective measurements against palatability (6).

Although there is a tendency for the slope of the curve expressing the logarithmic relation between s and t to relate to palatability, the use of sorption

* Surpasses 5% level of statistical significance.

technique as a means of determining quality is usually too cumbersome to be of practical significance (6). However, the results indicate that sorption of carbon dioxide bears some relation to milk powder structure as affected by drying practice and evident in the palatability of the reconstituted product. Attention is being given to possible use of this technique as a quantitative measure of milk powder quality.

Acknowledgments

The author wishes to express his thanks to Misses B. Metcalfe and J. Steacie for technical assistance during the course of this work.

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DRIED MILK POWDER

III. THE EFFECT OF LIGHT ON KEEPING QUALITY¹

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Abstract

At an average temperature of 6° C., exposure of both whole and skim milk powders to sunlight caused more rapid deterioration in quality than occurred in the dark. Ultra-violet light with a principal wave length of 3800 Å accelerated deterioration in whole milk powder stored at 38° C., but had no significant effect on skim milk powders; the effect of this light on whole milk powders was less pronounced than that produced by sunlight. Storage of samples at 38° C. under different light intensities indicated that the differences between ultra-violet and sunlight were the result of the difference in total energy of light falling upon the sample, rather than the difference in wave length of the activating light.

Introduction

One of the typical off-flavours known to develop in milk powder has been termed 'sunlight-taint.' Included in sunlight taint are other off-flavours developed as a result of exposure to bright artificial light (2, p. 437). The effect of light on numerous fats has been studied (3, pp. 139-152), but little definite information about the effect of sunlight on milk powder is available. The present paper describes an experiment designed to obtain some information about the effect of light on spray-dried milk powder of different fat levels and from different sources.

Materials and Methods

The milk powders used were the commercially available products of two Canadian companies. Plant X produced materials having 1, 26, 28, and 30% fat, and Plant Y produced products with 1, 26, and 28% fat. The skim milk powders (1% fat) had a moisture content of about 3%, while the whole milk powder had moisture contents of between 2.0 and 2.5%.

After the powders were tested for initial palatability, they were divided into four equal portions. One portion was subjected to outdoor sunlight for a period of 48 days, during which there was 215 hr. of sunlight. The average temperature during this period was 6° C. (43° F.). The 'light' energy falling on the samples was calculated as approximately 1.8 cal./sq. cm./min. A comparable set of samples in light-proofed containers was stored under the same conditions.

The third set was stored in the laboratory in proximity to an ultra-violet lamp for the periods shown in Fig. 1. The maximum light transmitted by this lamp was at a wave length of 3800 Å. and the range of ultra-violet produced was from 3200 to 4200 Å. The storage temperature in this phase

¹ Manuscript received June 13, 1945.

Contribution from the Division of Applied Biology, National Research Laboratories, Ottawa. Issued as Paper No. 141 of the Canadian Committee on Food Preservation and as N.R.C. No. 1326.

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of the experiment was 38° C. (100° F.). The calculated light energy falling on the sample was approximately 0.0034 cal./sq. cm./min. The fourth set of samples was stored in light-proofed containers at the same temperature to serve as controls.

An investigation of the effects of light intensity, done in duplicate, utilized only powder from one company (fat content of 1 and 26% and moisture content approximately 2.5%). The samples were spread in thin layers on large watch glasses and were protected from moisture change. Samples in both light-proofed and untreated watch glasses were stored (38° C.) at distances of one and four feet from a 100-watt incandescent lamp. The light intensities falling on the samples at the one-foot and four-foot levels were 0.0036 and 0.0004 cal./sq. cm./min., respectively. These materials were sampled for quality after three days (75 hr. exposure to light).

In all phases of this investigation the headspace gas in the containers was air.

The palatability of reconstituted samples was determined by the method used in these laboratories (6). Scoring was done on a scale from 10 to 0, 10 being the equivalent of excellent fresh whole or skim milk.

Results

The Effect of Light on Quality Changes During Storage

The results, using dry whole milk from two sources, and at different fat levels, were assessed by an analysis of variance. The effects shown to be significant are recorded in Fig. 1. In general, no differences in rate of deterioration were observed between powders from different sources or between powders

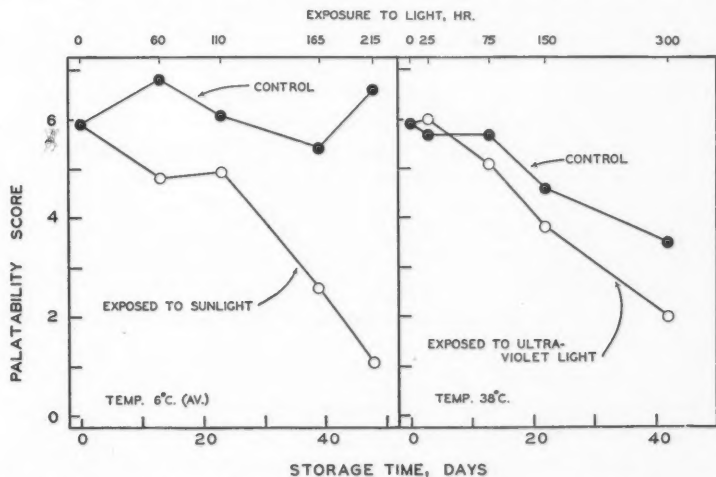


FIG. 1. The effect of sunlight and ultra-violet light on the deterioration of spray-dried whole milk powder.

with different fat levels. However, the 26% fat sample from Plant X appeared to be more light-sensitive than the others. The control samples held at 6° C. suffered no deterioration during the storage period, while control samples held at 38° C. deteriorated in a manner comparable to that noted earlier (5, 7).

The samples exposed to sunlight deteriorated much more rapidly than the control samples (Fig. 1). The samples subjected to 215 hr. of sunlight decreased about 4.8 palatability units. From the shape of the curves it is evident that the initial period of exposure to sunlight had little effect on palatability but the sunlight effects became more pronounced with increased exposure. Increased temperature accelerated the deterioration of these products (1). Since temperature and light effects are likely to be additive (3, pp. 139-152), it might be expected that sunlight effects would become more marked as the temperature increased. For these reasons, it is recommended that powders be exposed to sunlight for no longer than a five-hour period.

The difference in palatability between controls and samples exposed to the ultra-violet lamp was less marked than the differences between controls and samples exposed to sunlight. Exposure to 150 hr. illumination from this source decreased storage life by about 10 days, and after 300 hr. the difference between control and exposed powders was only 1.5 palatability units. Two factors may have been responsible for this effect. The energy for the ultra-violet lamp was extremely low as compared with sunlight, and in addition, the wave lengths emitted may not have been those causing maximum deterioration.

The skim milk powders (Table I) behaved in a manner similar to that previously noted (5), i.e., after a short period in storage the palatability seemed somewhat improved, suggesting that volatile degradation products formed in the powder during the dehydration process decreased during storage. This may not be noticeable in whole milk powders since fat is deteriorating simultaneously, the over-all result being lower palatability. Dried pork showed similar improvement in quality during the initial storage periods (4).

Only two effects of significance (Table I) were noted in these studies on skim milk. Powder from Source Y was generally of lower palatability than powder from Source X. This difference attained significance at the higher temperature. At both the combinations of temperature and light, the control samples received higher palatability scores than the exposed samples, but the difference was significant only in that portion of the experiment where powders were exposed to sunlight. The change in palatability of the controls and of the samples exposed to sunlight (average for samples from both sources) was as follows:

Storage time in days	0	13	23	39	48
Palatability (control)	2.2	3.6	5.0	4.3	5.6
Palatability (exposed)	2.2	3.1	3.6	2.5	2.2

The control samples showed some tendency to increase in palatability throughout the storage period, while the exposed samples increased slightly initially and then decreased.

TABLE I

THE EFFECT OF LIGHT, SOURCE OF POWDER, AND TIME OF EXPOSURE ON
THE QUALITY OF SPRAY-DRIED SKIM MILK

Storage conditions			
Sunlight (av. temp. 6° C.)		Ultra-violet (temp. 38° C.)	
Variable under study	Mean palatability	Variable under study	Mean palatability
Treatment		Treatment	
Exposed to light	2.8	Exposed to light	3.9
Control	4.6	Control	4.2
Source of powder		Source of powder	
X	4.0	X	4.6
Y	3.4	Y	3.5
Time		Time	
Storage (days), sunlight (hr.)		Storage (days), ultra-violet (hr.)	
0 0	2.2	0 0	2.2
13 60	3.4	3 25	4.7
23 110	4.2	13 75	3.9
39 165	3.4	22 150	3.5
48 215	3.9	42 300	4.0

Analysis of variance

Variance attributable to:	Degrees of freedom	Mean square	
		Sunlight	Ultra-violet light
Treatment	1	12.42*	0.25
Source of powder	1	1.38	5.52**
Time	3	0.75	0.91
Residual	10	1.26	0.52

* Exceeds 5% level of statistical significance.

** Exceeds 1% level of statistical significance.

Since skim milk of only 1% fat is affected by sunlight, it seems reasonable to assume that sunlight affected not only the fat fraction of the milk powder, but has some effect on the protein or carbohydrate components, or both.

The Effect of Light Intensity on Keeping Quality

The results of storing milk powders of 1% and 26% fat (initial palatabilities 6.9 and 6.3, respectively), under light of different intensities is shown in Table II. While the palatability scores applied at each trial differed significantly, the intensity of light falling on the sample caused significant differences in rate of deterioration. Skim milk powders did not deteriorate markedly except at the greatest light intensity, while whole milk powders were affected even by the light of lowest intensity, lending support to the previous observa-

TABLE II

THE EFFECT OF LIGHT ENERGY ON THE QUALITY OF SPRAY-DRIED WHOLE AND SKIM MILK POWDER STORED FOR THREE DAYS AT 38° C. (100° F.)

Table of means

Light energy affecting sample, cal./sq. cm./min.	Palatability scores			
	1% fat		26% fat	
	Trial 1	Trial 2	Trial 1	Trial 2
0	5.7	6.5	5.6	5.9
0.00038	5.9	6.6	3.8	5.1
0.0036	4.8	5.5	3.6	4.9

Analysis of variance

Source of variance	D. f.	Mean square
1% fat vs. 26% fat	1	31.01**
Between trials	2	11.04**
Light energy	2	15.02**
Light energy × fat levels	2	20.86**
Residual (Error I)	58	1.56
Tasters	9	3.80**
Residual (Error II)	45	0.64

** Exceeds 1% level of statistical significance.

tions (Fig. 1 and Table I). The high initial palatability of the skim milk was attributed to the fact that the container had been opened and was returned to storage for some time before the initiation of the experiment.

Since the light source used here produced a continuous spectrum from 2800 Å units far into the infra red, it was believed that the deterioration in relation to the energy expended might indicate whether sunlight taint of this product was a function of wave length or a function of the total energy. The results of calculations relating palatability decrease to light energy, shown in Fig. 2, seemed to indicate that deterioration was a function of wave length rather than the total energy expended on the sample. However, the palatability decrease of the control sample of whole milk powder subjected to the incandescent light was 2.5 times that occurring in the samples exposed to ultra-violet light. If comparable palatability decreases are assumed for both sets of control samples, the calculated decrease in the sample exposed to ultra-violet light (dotted curve in Fig. 2) almost equals the decrease in palatability occurring at corresponding light energy of the incandescent lamp. These calculations indicated that wave length of the incident light was a less important cause of deterioration than the energy expended on the samples.

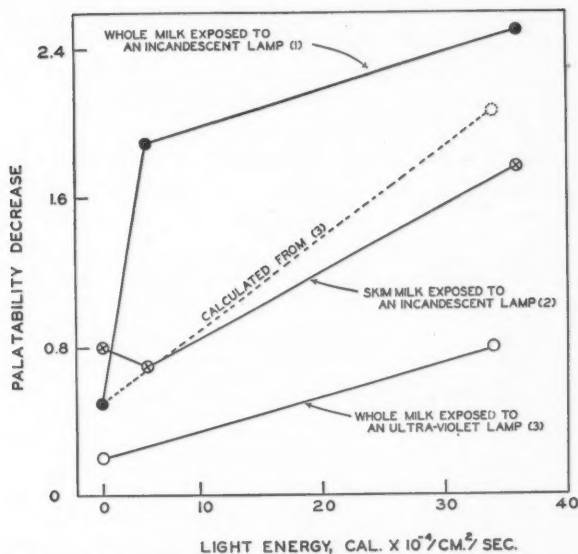


FIG. 2. The relation between light energy expended on the sample and palatability decrease. Dotted line shows values anticipated if milk powder exposed to the ultra-violet lamp had been as unstable as the powder exposed to the incandescent lamp.

Acknowledgments

The authors wish to express their thanks to Mr. F. W. Baker, meteorological observer, Dominion Department of Agriculture, Ottawa, and to Mr. A. S. Kennard, Canadian General Electric Company, Ottawa, for their kind assistance during this work.

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CANADIAN WILTSHIRE BACON

XXV. CHEMICAL PRESERVATIVES FOR MAINTAINING QUALITY AT HIGH STORAGE TEMPERATURES¹BY W. HAROLD WHITE², N. E. GIBBONS³, AND M. W. THISTLE⁴

Abstract

Seventy-seven chemical treatments were tested for their effectiveness in maintaining quality in bacon stored at 7.1°, 15.6°, and 23.8° C. for 30 days. The relative suitability of the compounds was assessed by organoleptic examination.

Treatments found to retard both slime formation and mould growth include magnesium benzoate with hydroxyacetic or citric acid; dimethylolurea; borobenzoic acid; acetylsalicylic acid; Aerosol-OS; Aseptex; Salol; cinnamic acid; and a mixture of benzoic acid, citric acid, salt, and oat flour and hulls. Several of the materials were relatively effective against bacteria but not against moulds, viz.: magnesium benzoate; sodium benzoate with hydroxyacetic acid; benzoic or boric acid with hydroxyacetic acid; cheesecloth treated with formaldehyde; and pyruvic acid. A few of the treatments, e.g. borax and Nacconal, retarded mould growth, but had little effect on bacteria. Because of possible toxicity or other considerations, none of the materials studied is considered to be entirely satisfactory.

Introduction

Under normal shipping conditions Canadian Wiltshire bacon sometimes arrived in England showing slime and other signs of incipient spoilage. The problem became more serious in the early war period because of unpredictable delays and the possible need for shipment in unrefrigerated space. Hence some precautionary measures were necessary to ensure that the bacon reached its destination in an edible and acceptable condition. Extensive investigations were carried out in these laboratories on the relative efficacy of smoking, of modifications in the curing process, and of chemical preservatives in maintaining the quality of bacon during longer holding periods, or at higher temperatures, than usual. Studies on smoking have been reported (8, 9, 10). The present paper gives the results of a study on the effectiveness of 77 chemical treatments in retarding spoilage of bacon stored at 7.1°, 15.6°, and 23.8° C.

The status of the use of preservatives in Canada has been ably discussed elsewhere and the literature on their application to fish products reviewed (5). This review and other recent investigations (1, 2, 3, 4, 7) have dealt mainly with the action of derivatives of benzoic acid. Since meat is more resistant to spoilage than fish and adequate refrigeration facilities are usually available,

¹ Manuscript received June 22, 1945.

Contribution from the Division of Applied Biology, National Research Laboratories, Ottawa. Issued as Paper No. 142 of the Canadian Committee on Food Preservation, and as N.R.C. No. 1327.

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there has been little incentive to search for chemical preservatives for this class of product. A recent review on the preservation of meat confirms this (6).

The British Ministry of Food early realized the necessity of providing some protection for bacon and authorized the use of borax or boric acid on Wiltshire sides*. Since these have relatively little preservative action, a search was made for more effective and less toxic chemicals. Accordingly, the value of most of the well-known preservatives was assessed, and many other compounds were investigated in an attempt to attain this objective. In the study of these hitherto untested compounds, secondary consideration was given to their toxicity since it was considered that ineffective substances could be discarded, while effective compounds, even if toxic, might give an indication of a suitable treatment. It is recognized that thorough toxicological studies would be required before any treatment could be considered for commercial adoption.

Materials and Procedure

Freshly cured Wiltshire backs were divided into five portions, each approximately 3 lb. in weight. The resulting samples were randomized amongst the various treatments and storage temperatures.

The preservatives studied, arranged where possible according to their chemical nature or type of treatment, are listed in Table II. For the most part, each preservative was tested at three concentrations for each of the storage temperatures, 7.1°, 15.6°, and 23.8° C. (45°, 60°, and 75° F.). The amount of preservative added was calculated as the percentage of the lean in an average sized back weighing 15 lb. and having 60% lean meat. On this basis, 0.8 gm. added to the 3-lb. test pieces approximated 0.1% of the lean meat. When the preservative was admixed with hydroxyacetic or citric acid, 0.8 gm. of the acid was thoroughly mixed with the desired weight of preservative. Depending on their physical condition, solid materials were either dusted or sprinkled onto the samples. Unless otherwise indicated, liquids were applied with a brush. When second and third applications of a liquid were made, sufficient time elapsed between successive treatments to permit at least superficial sorption of the preservative to occur. All preservatives were applied to the rib or top surfaces only of the lean meat. Each treated sample was wrapped in waxed, and overwrapped with brown, paper prior to storage.

Subjective examinations of the condition of each sample with respect to the surface colour, odour, slime formation, and mould growth for the lean meat were made after storage for 15 and 30 days.

Results

Assessment of the relative effectiveness of the various treatments from the data in the form of comments was difficult. An arbitrary system of scoring

*S. R. and O. for Foods, 1940, No. 547 (*Food*, 9 : 236. 1940).

quality was therefore adopted (Table I), based on the following considerations. The development of off-odours in the lean meat was believed to be the most important factor studied, since spoilage of the lean renders the bacon inedible. Surface growths of either bacteria or moulds detract from the appear-

TABLE I
SYSTEM OF SCORING EMPLOYED FOR ASSESSING THE RELATIVE SUITABILITY
OF PRESERVATIVES FOR WILTSHIRE BACON

Property	Score	Property	Score
Odour of the lean:		Mould:	
Good	3	None or slight	2
Slightly off or chemical	2	Medium	1
Definitely off	1	Heavy	0
Bad	0		
Slime:		Colour of the lean:	
None or slight	2	Acceptable	1
Medium	1	Poor	0
Heavy	0		

ance of the bacon, but, if not too far advanced and not accompanied by off-odours, they are considered to be of secondary importance since either can, for the most part, be removed readily by mechanical methods. The surface colour of the lean meat, as an attribute of quality, was ranked third in importance.

Quality scores for bacon, treated with the different amounts studied of the various preservatives and stored at 7.1°, 15.6°, and 23.8° C. (45°, 60°, and 75° F.) for 30 days, are given in Table II. It was considered that the above information gives sufficient indication of the relative effectiveness of the various treatments. Although presentation of other data, such as the detailed observations of the conditions of the samples, would be desirable, it is impractical because of the large number of treatments studied.

When bacon is stored without preservative for 30 days at 7.1° C., the odour of the lean is usually off and there may be some slime; at 15.6° C., the odour is definitely off and the surface is quite slimy; at 23.8° C., the meat is very bad.

For the most part, all treatments gave relatively good protection to the bacon stored at 7.1° C. Obvious differences were evident, however, at 15.6° C. and 23.8° C. The addition of citric or hydroxyacetic acid to the preservative usually had a beneficial effect. The use of hydroxyacetic acid appears to be preferable economically but, owing to its hygroscopic character, has the disadvantage of difficulty of application. It is also of some interest to note that in certain instances the preservative was more effective at 23.8° than at 15.6° C. The reason for this is not apparent.

Detailed examination of the total quality scores indicated that the following treatments were the most effective in retarding spoilage: benzoic acid with

TABLE II

QUALITY SCORES FOR BACON TREATED WITH VARIOUS PRESERVATIVES AND STORED AT 7.1°, 15.6°, AND 23.8° C. FOR 30 DAYS

No.	Preservative		Score ¹ at various storage temperatures		
	Treatment	Amount	7.1° C.	15.6° C.	23.8° C.
1	Benzoic acid	0.8 gm.	3220-7	1200-3	3200-5
		1.6 gm.	3220-7	0201-3	1200-3
		4.0 gm.	3221-8	1200-3	2220-6
2	Benzoic acid with citric acid	0.8 gm.	3221-8	1210-4	2210-5
		1.6 gm.	3221-8	2220-6	1210-4
		4.0 gm.	3221-8	3201-6	2220-6
3	Benzoic acid with hydroxyacetic acid	0.8 gm.	3221-8	3211-7	0210-3
		1.6 gm.	3221-8	3210-6	2211-6
		4.0 gm.	3220-7	3220-7	2221-7
4	Benzyl benzoate	Once	3220-7	1021-4	0220-4
		Twice	3220-7	1221-6	0010-1
		Thrice	3220-7	1020-3	0020-2
5	Ethyl benzoate	Once	2220-6	2210-5	1221-6
		Twice	2221-7	3211-7	3110-5
		Thrice	2221-7	3211-7	0101-2
6	Phenyl benzoate	0.8 gm.	2220-6	0220-4	1221-6
		1.6 gm.	2220-6	2220-6	2221-7
		4.0 gm.	2221-7	2221-7	2220-6
7	Methyl ester of <i>p</i> -hydroxybenzoic acid	0.4 gm.	2221-7	1210-4	1000-1
		0.8 gm.	2220-6	1220-5	2000-2
		1.6 gm.	2221-7	1220-5	2120-5
8	Ethyl ester of <i>p</i> -hydroxybenzoic acid	0.4 gm.	3221-8	0020-2	1100-2
		0.8 gm.	3220-7	2220-6	2220-6
		1.6 gm.	3221-8	2220-6	0220-4
9	Ethyl ester of <i>p</i> -hydroxybenzoic acid with citric acid	0.4 gm.	3221-8	3210-6	0220-4
		0.8 gm.	3221-8	2220-6	2200-4
		1.6 gm.	3221-8	2220-6	2200-4
10	Propyl ester of <i>p</i> -hydroxybenzoic acid	0.4 gm.	3221-8	1020-3	1010-2
		0.8 gm.	3221-8	1220-5	1120-4
		1.6 gm.	3221-8	0010-1	0020-2
11	Butyl ester of <i>p</i> -hydroxybenzoic acid	0.4 gm.	2221-7	0000-0	2220-6
		0.8 gm.	2021-5	0020-2	0020-2
		1.6 gm.	2220-6	0020-2	2220-6
12	Benzyl ester of <i>p</i> -hydroxybenzoic acid	0.4 gm.	2220-6	0021-3	0000-0
		0.8 gm.	3220-7	0121-4	0120-3
		1.6 gm.	3221-8	0120-3	0020-2
13	Sodium salt of methyl ester of <i>p</i> -hydroxybenzoic acid	0.4 gm.	2220-6	2220-6	0120-3
		0.8 gm.	2220-6	2220-6	0020-2
		1.6 gm.	2220-6	2220-6	3220-7

¹ The first digit indicates the rating assigned for the odour of the lean meat; the second and third for slime and mould, respectively; the fourth for the colour of the lean meat; and the fifth, the total score. Further details of the method of scoring are given in Table I.

TABLE II—Continued

QUALITY SCORES FOR BACON TREATED WITH VARIOUS PRESERVATIVES AND STORED
AT 7.1°, 15.6°, AND 23.8° C. FOR 30 DAYS—Continued

No.	Preservative		Score ¹ at various storage temperatures		
	Treatment	Amount	7.1° C.	15.6° C.	23.8° C.
14	Sodium salt of ethyl ester of <i>p</i> -hydroxybenzoic acid	0.4 gm.	3220-7	1220-5	0120-3
		0.8 gm.	2220-6	2200-4	2200-4
		1.6 gm.	2220-6	1220-5	0220-4
15	Sodium salt of propyl ester of <i>p</i> -hydroxybenzoic acid	0.4 gm.	3220-7	2120-5	0100-1
		0.8 gm.	3220-7	1220-5	1200-3
		1.6 gm.	3221-8	1200-3	3220-7
16	Sodium benzoate	0.8 gm.	2120-5	1101-3	0010-1
		1.6 gm.	3221-8	0101-2	0010-1
		4.0 gm.	3220-7	1211-5	1220-5
17	Sodium benzoate with citric acid	0.8 gm.	3221-8	1200-3	0000-0
		1.6 gm.	3221-8	2101-4	1100-2
		4.0 gm.	3221-8	2100-3	1100-2
18	Sodium benzoate with hydroxyacetic acid	0.8 gm.	3221-8	0221-5	3220-7
		1.6 gm.	3221-8	2220-6	3220-7
		4.0 gm.	3221-8	3220-7	0220-4
19	Magnesium benzoate	0.8 gm.	3221-8	0010-1	0100-1
		1.6 gm.	3221-8	0210-3	1210-4
		4.0 gm.	3221-8	3221-8	3220-7
20	Magnesium benzoate with citric acid	0.8 gm.	3220-7	3220-7	0110-2
		1.6 gm.	3220-7	2220-6	0100-1
		4.0 gm.	3221-8	3220-7	3220-7
21	Magnesium benzoate with hydroxyacetic acid	0.8 gm.	3221-8	0221-5	0010-1
		1.6 gm.	3221-8	3220-7	3200-5
		4.0 gm.	3220-7	3220-7	1210-4
22	Acetylsalicylic acid	1.6 gm.	2221-7	2221-7	2200-4
		4.0 gm.	3221-8	3220-7	2200-4
		8.0 gm.	3221-8	3220-7	3220-7
23	Phthalic acid with citric acid	0.8 gm.	3221-8	1200-3	0200-2
		1.6 gm.	3210-6	0201-3	0100-1
		4.0 gm.	1120-4	0221-5	1200-3
24	Sulphanilic acid with citric acid	0.8 gm.	3210-6	1100-2	0200-2
		1.6 gm.	3210-6	2200-4	0000-0
		4.0 gm.	3210-6	3210-6	0200-2
25	Dupont's No. IN-3102; 2% solution in petroleum ether	Once	3221-8	0021-3	0220-4
		Twice	2121-6	0020-2	0021-3
		Thrice	2220-6	0120-3	0020-2
26	Cinnamic acid with citric acid	0.8 gm.	3221-8	3220-7	3220-7
		1.6 gm.	3221-8	2220-6	3220-7
		4.0 gm.	3221-8	2220-6	2220-6

¹ The first digit indicates the rating assigned for the odour of the lean meat; the second and third for slime and mould, respectively; the fourth for the colour of the lean meat; and the fifth, the total score. Further details of the method of scoring are given in Table I.

TABLE II—Continued

QUALITY SCORES FOR BACON TREATED WITH VARIOUS PRESERVATIVES AND STORED
AT 7.1°, 15.6°, AND 23.8° C. FOR 30 DAYS—Continued

No.	Preservative		Score ¹ at various storage temperatures		
	Treatment	Amount	7.1° C.	15.6° C.	23.8° C.
27	Sodium sulphocarbolate	0.8 gm.	2221-7	0021-3	0221-5
		1.6 gm.	3221-8	0221-5	0120-3
		4.0 gm.	3221-8	0020-2	0021-3
28	Borobenzoic acid with citric acid	0.8 gm.	3221-8	2210-5	3200-5
		1.6 gm.	3221-8	3220-7	3220-7
		4.0 gm.	3220-7	3220-7	3220-7
29	Boric acid	1.6 gm.	3221-8	0100-1	2200-4
		4.0 gm.	3220-7	1201-4	2200-4
		8.0 gm.	3220-7	1201-4	2210-5
30	Boric acid with citric acid	1.6 gm.	3221-8	2211-6	1100-2
		4.0 gm.	3221-8	2201-5	1200-3
		8.0 gm.	3221-8	2221-7	1201-4
31	Boric acid with hydroxyacetic acid	0.8 gm.	3220-7	1210-4	3220-7
		1.6 gm.	3220-7	3220-7	0210-3
		4.0 gm.	3221-8	3221-8	2210-5
32	Boric anhydride with citric acid	0.8 gm.	3221-8	3210-6	2210-5
		1.6 gm.	3221-8	3211-7	1221-6
		4.0 gm.	3221-8	3221-8	0221-5
33	Borax	1.6 gm.	2220-6	1021-4	0021-3
		4.0 gm.	3220-7	0021-3	1121-5
		8.0 gm.	3220-7	0021-3	1121-5
34	Borax with citric acid	1.6 gm.	2220-6	1220-5	0110-2
		4.0 gm.	3220-7	2220-6	2220-6
		8.0 gm.	3220-7	1221-6	2221-7
35	Ethyl alcohol (95%); dipped 15 min.	—	2220-6	0200-2	3000-3
36	Ethyl alcohol (95%) with 2% hydroxyacetic acid; pH 3.35	Once	3200-5	0001-1	0220-4
		Twice	3210-6	0111-3	0001-1
		Thrice	3201-6	1010-2	0000-0
37	Ethylene glycol	Once	1021-4	0021-3	0001-1
		Twice	3020-5	0020-2	0101-2
		Thrice	1020-3	0020-2	0020-2
38	Propylene glycol	Once	2221-7	0020-2	0120-3
		Twice	3121-7	0021-3	0021-3
		Thrice	3121-7	0021-3	0020-2
39	Glycerol with hydroxyacetic acid; 50 gm. acid dissolved in 200 ml. water and added to 800 ml. glycerine; pH 1.8	Once	3220-7	3201-6	0101-2
		Twice	3111-6	3201-6	0001-1
		Thrice	3221-8	3200-5	3210-6

¹ The first digit indicates the rating assigned for the odour of the lean meat; the second and third for slime and mould, respectively; the fourth for the colour of the lean meat; and the fifth, the total score. Further details of the method of scoring are given in Table I.

TABLE II—Continued

QUALITY SCORES FOR BACON TREATED WITH VARIOUS PRESERVATIVES AND STORED
AT 7.1°, 15.6°, AND 23.8° C. FOR 30 DAYS—Continued

No.	Preservative		Score ¹ at various storage temperatures		
	Treatment	Amount	7.1° C.	15.6° C.	23.8° C.
40	Anhydrous sodium sulphate	4.0 gm.	1020-3	0221-5	0220-4
		8.0 gm.	3220-7	0021-3	0020-2
		16.0 gm.	3221-8	0220-4	0221-5
41	Anhydrous calcium chloride	4.0 gm.	3221-8	0020-2	1120-4
		8.0 gm.	3221-8	0010-1	0020-2
		16.0 gm.	3221-8	0001-1	0000-0
42	Oil of birch	Once	2220-6	2220-6	2220-6
		Twice	2220-6	2220-6	3200-5
		Thrice	2121-6	2220-6	2000-2
43	Oil of birch tar	Once	3120-6	0020-2	2020-4
		Twice	2120-5	2010-3	2020-4
		Thrice	2220-6	2021-5	2020-4
44	Oil of pine	Once	2221-7	2120-5	2120-5
		Twice	2220-6	2220-6	2220-6
		Thrice	2221-7	2121-6	2020-4
45	Oil of tar	Once	2220-6	2110-4	3210-6
		Twice	2220-6	2210-5	2210-5
		Thrice	2220-6	2220-6	2110-4
46	Essence of smoke	Once	3210-6	3210-6	3210-6
		Twice	3220-7	2100-3	3200-5
		Thrice	3220-7	2110-4	3210-6
47	Sawdust	Excess	3221-8	0221-5	3221-8
48	Wiping with rag treated with 1% formaldehyde solution	—	3021-6	0020-2	0000-0
49	Wiping with rag treated with glacial acetic acid	—	3221-8	2201-5	3201-6
50	Wiping with rag treated with saturated solution of benzoic acid containing 2% hydroxyacetic acid	—	2220-6	0220-4	0000-0
51	Dipping in aqueous solution of formaldehyde for one minute	2%	3220-7	3201-6	1100-2
		5%	3220-7	0110-2	0110-2
52	Wrapping in cheesecloth treated with formaldehyde	1%	3221-8	0111-3	0001-1
		3%	3220-7	0111-3	3211-7
		5%	3221-8	0020-2	3201-6
53	Sodium propionate	1.6 gm.	3221-8	2210-5	0010-1
		4.0 gm.	2221-7	2011-4	0020-2
		8.0 gm.	2221-7	2210-5	1110-3

¹ The first digit indicates the rating assigned for the odour of the lean meat; the second and third for slime and mould, respectively; the fourth for the colour of the lean meat; and the fifth, the total score. Further details of the method of scoring are given in Table I.

TABLE II—Continued

QUALITY SCORES FOR BACON TREATED WITH VARIOUS PRESERVATIVES AND STORED
AT 7.1°, 15.6°, AND 23.8° C. FOR 30 DAYS—Continued

No.	Preservative		Score ¹ at various storage temperatures		
	Treatment	Amount	7.1° C.	15.6° C.	23.8° C.
54	Sodium propionate with citric acid	1.6 gm.	3220-7	2200-4	1101-3
		4.0 gm.	2220-6	1200-3	2210-5
		8.0 gm.	2221-7	2221-7	1101-3
55	Urotropin	1.6 gm.	2220-6	0000-0	1100-2
		4.0 gm.	3111-6	2201-5	2100-3
		8.0 gm.	3220-7	2200-4	2200-4
56	Nickel pectinate (flake)	0.8 gm.	2221-7	0220-4	0220-4
		1.6 gm.	3220-7	0220-4	0220-4
		4.0 gm.	1220-5	0020-2	0120-3
57	Sodium pyrophosphate with hydroxyacetic acid	0.8 gm.	3220-7	1221-6	0201-3
		1.6 gm.	3121-7	0220-4	0221-5
		4.0 gm.	3221-8	0220-4	0221-5
58	Sulphamic acid with citric acid	0.8 gm.	3221-8	1201-4	1220-5
		1.6 gm.	3211-7	3200-5	0200-2
		4.0 gm.	0220-4	3220-7	1200-3
59	Pyruvic acid	Once	3220-7	3211-7	3200-5
		Twice	3220-7	3210-6	3211-7
		Thrice	3221-8	3220-7	3201-6
60	Hydroxyacetic acid	0.8 gm.	3221-8	0220-4	0010-1
		1.6 gm.	3221-8	0121-4	0000-0
		4.0 gm.	3221-8	0101-2	3200-5
61	Citric acid	0.8 gm.	3200-5	0100-1	0001-1
		1.6 gm.	3121-7	1000-1	2201-5
		4.0 gm.	3210-6	1100-2	0100-1
62	Hydrochloric acid; dipped 15 min.	0.05 <i>N</i>	3210-6	2200-4	0000-0
		0.1 <i>N</i>	2220-6	0200-2	0000-0
		0.5 <i>N</i>	2201-5	1200-3	2100-3
63	Zephirin; dipped 15 min.	1 : 5000	2011-4	0010-1	0000-0
		1 : 10,000	2120-5	0011-2	0000-0
		1 : 20,000	2220-6	0000-0	0100-1
64	Moldex	0.8 gm.	3221-8	1220-5	2220-6
		1.6 gm.	3220-7	2221-7	2220-6
		4.0 gm.	2220-6	2220-6	2220-6
65	Aseptex	0.8 gm.	3220-7	2111-5	0210-3
		1.6 gm.	3220-7	3201-6	0200-2
		4.0 gm.	3120-6	3220-7	3220-7
66	Salol	0.8 gm.	3220-7	0020-2	2220-6
		1.6 gm.	2221-7	2220-6	1121-5
		4.0 gm.	2220-6	2221-7	2220-6

¹ The first digit indicates the rating assigned for the odour of the lean meat; the second and third for slime and mould, respectively; the fourth for the colour of the lean meat; and the fifth, the total score. Further details of the method of scoring are given in Table I.

TABLE II—*Concluded*

QUALITY SCORES FOR BACON TREATED WITH VARIOUS PRESERVATIVES AND STORED
AT 7.1°, 15.6°, AND 23.8° C. FOR 30 DAYS—*Concluded*

No.	Preservative		Score ¹ at various storage temperatures		
	Treatment	Amount	7.1° C.	15.6° C.	23.8° C.
67	Aerosol-OS	0.8 gm.	3221-8	1121-5	1221-6
		1.6 gm.	3221-8	0021-3	3221-8
		4.0 gm.	3221-8	3221-8	3221-8
68	Nacconal	0.8 gm.	3220-7	2221-7	0120-3
		1.6 gm.	3220-7	3221-8	0020-2
		4.0 gm.	3221-8	3220-7	2220-6
69	Allantoin	0.8 gm.	3221-8	0220-4	0021-3
		1.6 gm.	3221-8	0021-3	0220-4
		4.0 gm.	3220-7	0021-3	0021-3
70	Dimethylol urea	1.6 gm.	3220-7	3211-7	3220-7
		3.2 gm.	3220-7	3220-7	3210-6
		8.0 gm.	3220-7	3220-7	3220-7
71	Sodium hydroxide (flake)	1.0 gm.	3220-7	0221-5	0220-4
		4.0 gm.	3220-7	3020-5	3220-7
		7.0 gm.	3220-7	3020-5	3220-7
		10.0 gm.	3220-7	3020-5	3220-7
72	Oat hulls	Excess	3220-7	0221-5	0221-5
73	Sodium bicarbonate and citric acid; packed in oat hulls; NaHCO ₃ : citric acid = 252:192	10 gm.	3221-8	0221-5	0201-3
		20 gm.	3221-8	2221-7	0221-5
		30 gm.	3220-7	0221-5	0221-5
74	Sodium bicarbonate and hydroxyacetic acid; packed in oat hulls; NaHCO ₃ : CH ₃ OH.COOH = 84:76	10 gm.	3221-8	0221-5	0210-3
		20 gm.	3221-8	2221-7	0211-4
		30 gm.	3221-8	2221-7	0210-3
75	Benzoic acid (4 parts), citric acid (2 parts), sodium chloride (20 parts), oat flour (46 parts), and oat hulls (28 parts)	Excess	3221-8	3221-8	3221-8
76	Surface of meat dried and scorched by a gas torch	—	3220-7	0000-0	2110-4
77	Sodium pyrophosphate	0.8 gm.	3221-8	3221-8	0020-2
		1.6 gm.	3221-8	0020-2	0021-3
		4.0 gm.	3221-8	0220-4	0021-3

¹ The first digit indicates the rating assigned for the odour of the lean meat; the second and third for slime and mould, respectively; the fourth for the colour of the lean meat; and the fifth, the total score. Further details of the method of scoring are given in Table I.

hydroxyacetic acid; phenyl benzoate; sodium benzoate with hydroxyacetic acid; magnesium benzoate with and without hydroxyacetic acid; acetylsalicylic acid; cinnamic acid with citric acid; borobenzoic acid with citric acid; boric acid with hydroxyacetic acid; glycerol with hydroxyacetic acid;

packing in sawdust; wrapping in cheesecloth treated with formaldehyde; pyruvic acid; Aseptex; Salol; Aerosol-OS; dimethylolurea; sodium hydroxide; and a mixture of benzoic acid, citric acid, sodium chloride, and oat flour and hulls.

A group of treatments of an intermediate degree of effectiveness included: benzoic acid with citric acid; benzyl and ethyl benzoates; ethyl ester of *p*-hydroxybenzoic acid with citric acid; propyl and benzyl esters of *p*-hydroxybenzoic acid; sodium salts of methyl, ethyl, and propyl esters of *p*-hydroxybenzoic acid; magnesium benzoate with citric acid; boric acid with and without citric acid; boric anhydride with citric acid; borax with and without citric acid; dipping for 15 min. in ethyl alcohol; oils of birch, birch tar, pine, and tar, essence of smoke; wiping with rags treated with glacial acetic acid; dipping for one minute in an aqueous solution of formaldehyde; sodium propionate with and without citric acid; urotropin; Moldex; and Nacconal.

Several treatments were found to be quite unsuitable for bacon, viz.: benzoic acid; methyl, ethyl, and butyl esters of *p*-hydroxybenzoic acid; sodium benzoate with and without citric acid; phthalic or sulphanilic acid with citric acid; 2% solution of Dupont's No. IN-3102 in petroleum ether; sodium sulphocarbolate; 95% ethyl alcohol with 2% hydroxyacetic acid; ethylene or propylene glycol; anhydrous sodium sulphate or calcium chloride; wiping with rags treated with 1% formaldehyde solution or with a saturated solution of benzoic acid containing 2% hydroxyacetic acid; nickel pectinate; sodium pyrophosphate with and without hydroxyacetic acid; sulphamic acid with citric acid; hydroxyacetic or citric acids; dipping for 15 min. in hydrochloric acid or in Zephirin; allantoin; oat hulls; dusting with a mixture of sodium bicarbonate with citric or hydroxyacetic acid and then packing in oat hulls; drying and scorching the surface of the meat with a gas torch.

Examination of the data on slime and mould formation indicates that very few of the treatments were effective at the lowest concentration studied. At the highest concentration several treatments were quite effective in retarding both slime formation and mould growth, namely, magnesium benzoate with hydroxyacetic or citric acid; borobenzoic acid; acetylsalicylic acid; Aerosol; Aseptex; Salol; cinnamic acid; dimethylolurea; and a mixture of benzoic acid, citric acid, salt, and oat flour and hulls. Several of the materials were relatively effective against bacteria but not against moulds, viz.: sodium benzoate, phthalic acid or boric acid with citric acid; glycerol with hydroxyacetic acid; essence of smoke; and pyruvic acid. A few of the treatments retarded mould growth, but had little effect on bacteria, e.g. borax, ethylene or propylene glycol, and benzyl benzoate.

Discussion

The "ideal" preservative for bacon would be one that is non-toxic, even if ingested repeatedly, and that would prevent the growth of bacteria and moulds and the development of rancidity. It should have no effect on the colour, flavour, odour, or texture of the meat. The method of application

should fit into normal plant practice, and the cost should be as reasonable as possible. None of the preservatives studied here can be considered to satisfy all of these criteria. Investigations on these and other treatments are being continued.

Acknowledgments

The authors wish to express their appreciation to Mr. T. A. Steeves, formerly Refrigeration Engineer, National Research Laboratories, for establishing and maintaining the storage conditions, and to L. Moore and R. Moore, Laboratory Assistants, for their assistance with the investigations.

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CANADIAN WILTSHIRE BACON

XXVI. FURTHER OBSERVATIONS ON THE PRESERVATION OF QUALITY AT HIGH STORAGE TEMPERATURES¹BY W. HAROLD WHITE², M. W. THISTLE³, AND T. A. STEEVES⁴

Abstract

The suitability of polyvinyl alcohols, dimethylol urea, commercial wax dispersions, carbon dioxide, sodium hydroxide, and a combined smoking and partial desiccation treatment as preservatives for bacon stored at 15.6° C. (60° F.) was investigated. Dimethylol urea was a relatively effective preservative and mouldicide for smoked bacon. Of the methods studied for applying this material, a vaseline paste was most suitable, but not entirely satisfactory. Back bacon, smoked for six hours at 40.6° C. (105° F.), and subsequently dried at this temperature to moisture contents of about 55 to 60% or less, was in a satisfactory condition, except for a heavy mould growth, after 60 days' storage. Sodium hydroxide was relatively ineffective when applied as an aqueous solution. Polyvinyl alcohols, wax dispersions, and carbon dioxide were unsuitable under the conditions studied.

Introduction

Extensive investigations have been made in these laboratories on methods for the prevention of spoilage of bacon during storage at high temperatures, of which the effectiveness of smoking (4, 8, 9), strong curing (6), and a number of chemical treatments (7) have been described previously. The present paper deals with further observations made on chemical preservatives. In addition, the effect of partial desiccation on the keeping quality of bacon is described.

Polyvinyl Alcohols

The properties of the polyvinyl alcohols would appear to make these materials worthy of investigation as protective coverings for food products. Being water-soluble, they may be directly applied by immersion of the food in an aqueous solution of the alcohol, thus avoiding the use of volatile solvents normally required for most synthetic plastics, and the consequent possible tainting of the foodstuff. Moreover, it has been stated that polyvinyl alcohol films are resistant to both moulds and bacteria (1, p. 2). Their use in the preservation of shell eggs has been described (2).

A preliminary experiment was made to ascertain the relative suitability of Types A and B polyvinyl alcohols (1), the concentration required to give a satisfactory film, and the effect of varying the immersion time of bacon in the polyvinyl alcohol. For this purpose, two thin slices of Wiltshire back bacon

¹ Manuscript received April 23, 1945.

Contribution from the Division of Applied Biology, National Research Laboratories, Ottawa, Canada. Issued as Paper No. 143 of the Canadian Committee on Food Preservation and as N.R.C. No. 1328.

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were dipped for each of one and three minutes in the following solutions of polyvinyl alcohol*: Type A, low (No. 623) and medium viscosity (No. 488), 2, 6, 10, 14, and 18%; Type B, medium viscosity (No. 349), 2, 6, 10, 14, and 18%; and Type B, high viscosity (No. 391), 2, 6, and 10%. (All concentrations are expressed as gm. of polyvinyl alcohol per 100 ml. of water.) After dipping, the slices were permitted to drain and dry at laboratory temperature.

Visual examination indicated that the Type B polyvinyl alcohols gave much superior films to those of Type A at all concentrations, and that the most desirable concentration of the Type B polyvinyl alcohols was 14% for the medium viscosity (No. 349) and 10% for the high viscosity (No. 391) polymers. There was no evident difference between samples dipped for one and three minutes.

With this preliminary information available a study was made of the effectiveness of Type B polyvinyl alcohol as a preservative for Wiltshire bacon. Two backs, removed from the right and left sides of the same hog, were each cut into nine pieces. Two pieces were allocated at random to each of the following treatments with medium viscosity (No. 349) polyvinyl alcohol: (1) 7% aqueous solution; (2) 10% aqueous solution; (3) 14% aqueous solution; (4) 7% aqueous solution; hydrogen ion concentration adjusted acid to methyl red; (5) 14% aqueous solution; hydrogen ion concentration adjusted acid to methyl red; (6) 7% aqueous solution containing 0.2 parts dimethylol urea to one part of polyvinyl alcohol and hydrogen ion concentration adjusted acid to methyl red; (7) as for (6), but 10% aqueous solution of polyvinyl alcohol; (8) as for (6), but 14% aqueous solution of polyvinyl alcohol; and (9) control.

The small cuts of bacon were dipped in the appropriate solution at room temperature for one minute, drained, and dried in a rapid current of air for approximately one hour. This procedure was repeated, thus giving each sample a double dip. All samples were left for 20 hr. at room temperature to permit drying of the polyvinyl alcohol, wrapped in waxed paper and over-wrapped in brown, and stored at 15.6° C. (60° F.) for a period of one month.

The experiment was repeated with the high viscosity (No. 391) polyvinyl alcohol as described above except that concentrations of 6, 9, and 12% were used.

Although it had been planned originally to make detailed chemical and bacteriological examinations of the samples at the end of the storage period, it was found that their condition did not merit more than visual inspection. All samples were putrid, slimy, and mouldy. However, the condition of the treated samples appeared to be slightly better than that of the controls. Medium viscosity polyvinyl alcohol appeared to give slightly more protection than the high viscosity. The addition of acid or dimethylol urea to the polyvinyl alcohol solution had no apparent effect. It may be concluded that the polyvinyl alcohol treatments employed here were unsatisfactory for the preservation of bacon at high storage temperatures.

* The selection of polyvinyl alcohols studied was based on recommendations of Canadian Industries Limited, Montreal.

Dimethylol Urea

The results of previous investigations (4, 8) and of trial shipments to England (5) showed that smoking caused an accelerated growth of moulds on bacon but gave marked protection from other forms of spoilage. In a previous study on chemical preservatives (7), it was observed that dimethylol urea not only retarded putrefaction in unsmoked bacon stored at high temperatures, but prevented mould growth. Accordingly, further studies were made on the application of dimethylol urea to smoked bacon.

Use of Dimethylol Urea on Smoked Bacon

The effect of dimethylol urea on mould growth on smoked bacon was determined for a freshly cured Wiltshire back that had been smoked at an air temperature of 62.8° C. (145° F.) to an internal meat temperature of 51.7° C. (125° F.), and subsequently divided into eight approximately equal portions. Two pieces were allocated at random to each of the following: treatment with 2, 5, and 10 gm. of dimethylol urea dusted on the rib and meat surfaces and no treatment (control samples).

After treatment each sample was wrapped in waxed, and overwrapped with brown, paper and stored at 15.6° C. (60° F.) for one month. An organoleptic examination of the samples was made at the end of the storage period.

The results of the examination of the samples are given in Table I. The general condition of the meat treated with the greater amounts of dimethylol

TABLE I

RESULTS OF ORGANOLEPTIC EXAMINATION OF SMOKED BACON TREATED WITH DIMETHYLOL UREA AND STORED AT 15.6° C. FOR ONE MONTH

Wt. of dimethylol urea, gm.	Sample No.	Lean				Fat	
		Colour	Odour	Slime	Mould	Colour	Odour
2	1	Good	Good	None	Heavy	Good	Good
	2	Good	Good	None	Heavy	Good	Good
5	3	Good	Excellent	None	None	Good	Good
	4	Good	Excellent	None	Heavy	Good	Good
10	5	Good	Excellent	None	None	Good	Good
	6	Good	Excellent	None	Slight	Good	Good
Control	7	Good	Fair	None	Heavy	Good	Good
	8	Good	Fair	None	Heavy	Good	Good

urea was excellent. There was every indication that, when the surface was adequately covered, dimethylol urea prevented the growth of moulds on smoked bacon. The somewhat variable results obtained with the use of 5 and 10 gm. of dimethylol urea are attributed to unequal distribution of the material on the surface.

Methods of Application of Dimethylol Urea to Smoked Bacon

In the previous experiment it was shown that dimethylol urea retarded both bacterial and mould growth on bacon if the surface was adequately covered with the material. The object of the present experiment was to determine the most suitable of several possible methods for applying dimethylol urea to bacon.

One freshly smoked Wiltshire back was divided into 10 approximately equal portions. Two pieces were allocated at random to each of five treatments: dimethylol urea as (1) a water paste; (2) a water dip; (3) a water spray; (4) a mineral oil dispersion; and (5) a vaseline paste. The various amounts of chemical added are noted in Table II. Dipped or sprayed samples were allowed to drain for 15 min. Pastes were applied manually. After treatment, the samples were wrapped in waxed paper and overwrapped in brown. Organoleptic examination of the condition of the bacon was made after 30, 45, 60, and 78 days' storage at 15.6° C.

TABLE II

QUANTITIES OF DIMETHYLOL UREA APPLIED BY VARIOUS METHODS TO SMOKED BACON

Treatment medium	Sample No.	Wt. of sample, gm.	Wt. of added material, gm.	Dimethylol urea in added material, %	Dimethylol urea added, gm.
Water paste	1	556	101	55	56
	2	440	87	55	48
Water dip	3	467	59	45	27
	4	458	65	45	29
Water spray	5	445	26	45	12
	6	469	23	45	10
Mineral oil	7	595	171	42	72
	8	465	111	42	47
Vaseline paste	9	501	160	35	56
	10	455	166	35	58

The results obtained for 30 and 78 days' storage are given in Table III. While large amounts of dimethylol urea in a water paste prevented mould development for 78 days, abnormal desiccation of the meat was observed even after 30 days' storage. The water dip and spray treatments were the least effective of those studied. The mineral oil paste was unsuitable because of excessive oiliness of the bacon and packaging materials. The samples receiving the vaseline paste treatment retained a fairly good external appearance throughout storage. No slime was evident on any of the samples. The internal condition of the meat treated with the vaseline paste was markedly superior to that of all other samples (Table IV). It would appear that the addition of dimethylol urea in a vaseline paste was preferable to the other methods of application studied in retaining the over-all quality of bacon. However, none of the treatments is considered to be entirely satisfactory.

TABLE III

RESULTS OF ORGANOLEPTIC EXAMINATION OF SMOKED WILTSHIRE BACON STORED AT 15.6° C. AND TREATED WITH DIMETHYLOL UREA BY VARIOUS METHODS

Treatment medium	Days in storage	Sample No.	Lean				Fat		Comments
			Colour	Odour	Slime	Mould	Colour	Odour	
Water paste	30	1	Good	Good	None	None	Good	Good	Fairly dry
		2	Sl. dark	Sl. chem.	None	None	Good	Good	Fairly dry
	78	1	Dark; greyed	Sl. chem.	None	None	Good	Good	Very dry
		2	Dark; greyed	Sl. chem.	None	None	Good	Good	Very dry
Water dip	30	3	Sl. dark	Sl. chem.	None	One colony	Good	Good	Dry
		4	Sl. dark	Sl. chem.	None	None	Good	Good	Dry
	78	3	Dark; greyed	Sl. chem.	None	Heavy	Good	Mouldy	Very dry
		4	Dark; greyed	Sl. chem.	None	Heavy	Good	Mouldy	Very dry
Water spray	30	5	Sl. dark	Sl. chem.	None	None	Good	Good	Dry
		6	Sl. dark	Sl. chem.	None	None	Good	Good	Dry
	78	5	Dark; greyed	Sl. chem.	None	Heavy	Good	Mouldy	Very dry
		6	Dark; greyed	Sl. chem.	None	Heavy	Good	Mouldy	Very dry
Mineral oil	30	7	Sl. dark	Good	None	None	Good	Good	Oily
		8	Sl. dark	Good	None	None	Good	Good	Oily
	78	7	Dark; greyed	Sl. chem.	None	None	Sl. dark	Good	Oily
		8	Dark; greyed	Sl. chem.	None	Few	Sl. dark	Good	Oily
Vaseline paste	30	9	Good	Good	None	Very slight	Good	Good	Moist
		10	Good	Good	None	None	Good	Good	Moist
	78	9	Sl. brown*	Sl. chem.	None	Slight	Good	Good	Slightly dry
		10	Sl. brown*	Sl. chem.	None	Slight	Good	Good	Slightly dry

*Darkening of the meat indicating slight methaemoglobin formation.

TABLE IV

RESULTS OF ORGANOLEPTIC EXAMINATION OF CUT SURFACE OF SMOKED WILTSHIRE BACON AFTER TREATMENT WITH DIMETHYLOL UREA, AND STORAGE FOR 78 DAYS AT 15.6° C.

Treatment medium	Sample No.	Cut meat surface			Remarks
		Colour	Odour	Moisture	
Water paste	1	Dull, unattractive	Chemical	Very dry	Mould infiltration
	2	Dull, unattractive	Chemical	Very dry	Mould infiltration
Water dip	3	Dull, poor	Chemical	Very dry	Mould infiltration
	4	Dull, poor	Chemical	Very dry	Mould infiltration
Water spray	5	Dull, poor	Chemical	Very dry	Interior very dry
	6	Dull, poor	Chemical	Very dry	Interior very dry
Mineral oil	7	Good	Sl. chemical	Sl. dry	Appearance, too oily and unattractive.
	8	Good	Sl. chemical	Sl. dry	Appearance, too oily and unattractive
Vaseline paste	9	Excellent	Very good	Excellent	Interior very well preserved and attractive
	10	Excellent	Very good	Excellent	Appearance, very well preserved and attractive

Wax Dispersions and Dimethylol Urea as Preservatives for Bacon

Two wax dispersions*, referred to as *A* and *B*, were investigated with respect to their preservative effect on bacon when used both alone and in conjunction with dimethylol urea. For this purpose an unsmoked, Wiltshire-cured back was divided into 10 approximately equal portions. Two pieces were allocated at random to each of five treatments: (1) dimethylol urea; (2) Wax Dispersion *A*; (3) Wax Dispersion *B*; (4) dimethylol urea followed by *A*; and (5) dimethylol urea followed by *B*. The dimethylol urea was applied by dipping the bacon in a 45% aqueous slurry, and the wax dispersions, with a spray gun. The samples were drained for 15 min., then wrapped in waxed paper, and overwrapped in brown. Organoleptic examination of the condition of the bacon was made after 15 and 31 days' storage at 15.6° C.

The results are given in Table V. Both wax dispersions failed to protect the meat from spoilage and mould growth. Moreover, the fat developed off-odours and became discoloured. In marked contrast, dimethylol urea gave excellent protection from both bacterial and mould growth. The addition of wax dispersions to dimethylol urea had no apparent supplemental effect.

Carbon Dioxide

It was considered that spoilage of bacon stored at high temperatures might be retarded if treated with a suitable chemical mixture to effect a slow evolution of carbon dioxide gas. In an initial experiment a mixture of 25 gm. of citric acid and 33 gm. of sodium bicarbonate was dusted on the rib and meat surfaces of a piece of back bacon about 4 in. in length. The treated sample was stored in a desiccator at room temperature. It was observed that rather vigorous reaction occurred, and little or no preservative effect was obtained. An attempt was made to retard the reaction by covering the meat surface with about 10 gm. of wood sawdust prior to the addition of the acid-bicarbonate mixture. A measure of preservative action was obtained, but not sufficient to be of practical value.

In a second experiment bacon was stored in latex rubber bags containing carbon dioxide. For this purpose a Wiltshire-cured back was divided into 10 equal portions, each of about 675 gm. in weight. Two pieces were allocated at random to each of the following treatments with solid carbon dioxide: (1) 20 gm.; (2) 15 gm.; (3) 10 gm.; (4) 5 gm.; and (5) control. The appropriate sample was placed in an expanded latex bag, the desired amount of solid carbon dioxide added, and the bag evacuated and sealed. The samples were inspected after 15 and 30 days' storage at 15.6° C.

The results for the organoleptic examination are given in Table VI. None of the concentrations of carbon dioxide employed prevented the spoilage of bacon. The presence of carbon dioxide not only had no apparent inhibitive effect on slime formation, but stimulated the growth of moulds. This latter

* Du Pont Chemicals No. A-1199 and A-1200, respectively. These were kindly supplied by Canadian Industries Limited, Montreal.

TABLE V
RESULTS OF ORGANOLEPTIC EXAMINATION OF WILTSHIRE BACON TREATED WITH DIMETHYLOL UREA AND
WAX DISPERSIONS, AND STORED AT 15.6° C.

Treatment	Days in storage	Sample No.	Lean				Fat	
			Colour	Odour	Slime	Mould	Colour	Odour
Dimethylol urea	15	1	Sl. light	Sl. chem.	Very moist	None	Satisfactory	Satisfactory
	31	2	Sl. light	Sl. chem.	Very moist	None	Satisfactory	Satisfactory
		1	Sl. brown*	Sl. chem.	None	None	Satisfactory	Satisfactory
		2	Sl. brown*	Sl. chem.	None	None	Satisfactory	Satisfactory
Wax Dispersion A	15	3	Good	Sl. off	None	Medium	Yellow	—
		4	Too red	Bad	Slight	Medium	—	—
	31	3	Too bright; mottled	Bad	Medium	Heavy	Yellow	—
		4	Too bright; mottled	Bad	Medium	Heavy	Dark	Off
Wax Dispersion B	15	5	Pink patches	Sl. off	Very sl. moist	One colony	—	—
		6	Pink patches	Very sl. off	Very sl. moist	One colony	—	—
	31	5	Pinkish	Bad	Slight	Medium	Discoloured	Off
		6	Pinkish	Bad	Slight	Slight	Discoloured	Off
Dimethylol urea followed by Wax Dispersion A	15	7	Sl. light	Sl. chem.	Moist	None	Satisfactory	Satisfactory
		8	Sl. dark	Sl. chem.	Moist	None	Satisfactory	Satisfactory
	31	7	Sl. brown*; very sl. mauve	Sl. chem.	None	None	Satisfactory	Satisfactory
		8	Med. brown*; sl. mauve and dark	Sl. chem.	None	None	Satisfactory	Satisfactory
Dimethylol urea followed by Wax Dispersion B	15	9	Light mauve	Sl. chem.	Moist	None	Satisfactory	Satisfactory
		10	Light mauve	Sl. chem.	Moist	None	Satisfactory	Satisfactory
	31	9	Sl. brown*; sl. grey	Sl. chem.	None	None	Satisfactory	Satisfactory
		10	Med. brown*; sl. grey	Sl. chem.	None	None	Satisfactory	Satisfactory

*Darkening of the meat indicating slight melhaemoglobin formation.

TABLE VI

RESULTS OF ORGANOLEPTIC EXAMINATION OF WILTSHIRE BACON TREATED WITH CARBON DIOXIDE AND STORED IN LATEX BAGS AT 15.6° C.

Wt. of CO ₂ , gm.	Days in storage	Sample No.	Lean				Fat	
			Colour	Odour	Slime	Mould	Odour	Colour
0	15	1	Sl. bright	—	—	None	—	Sl. stained
		2	Sl. bright	—	—	None	—	Sl. stained
5	30	1	Good	Sl. off	Heavy	None	Good	Sl. stained
		2	Good	Sl. off	Heavy	None	Good	Good
	15	3	Sl. brown*	—	—	Slight	—	Good
		4	Sl. brown*	—	—	Slight	—	Good
10	30	3	Sl. brown*	Off	Heavy	Slight	Good	Good
		4	Sl. brown*	Sl. off	Heavy	Slight	Good	Good
	15	5	Bright	—	—	Medium	—	Good
		6	Bright	—	—	Slight	—	Good
15	30	5	Good	Sl. off	Heavy	Slight	Good	Good
		6	Good	Sl. off	Heavy	Slight	Good	Good
	15	7	Bright	—	—	Medium	—	Good
		8	Bright	—	—	Heavy	—	Good
20	30	7	Sl. brown*	Sl. off	Heavy	Heavy	Good	Good
		8	Sl. brown*	Sl. off	Heavy	Slight	Good	Good
	15	9	Bright	—	—	Very slight	—	Good
		10	Bright	—	—	Medium	—	Good
30		9	Good	Bad	Heavy	Medium	Good	Good
		10	Good	Sl. off	Heavy	Medium	Good	Good

*Darkening of the meat indicating slight methaemoglobin formation.

effect is presumably attributable to a lowering of the pH of the meat because of solution of carbon dioxide. The results of this and the preceding experiment indicate that carbon dioxide is not effective in preventing spoilage of bacon stored at high temperatures.

Sodium Hydroxide

In a previous experiment on the use of sodium hydroxide as a preservative for bacon (7) the compound was applied in the solid form. Because of its hygroscopic nature, however, considerable difficulty was encountered in applying and distributing the hydroxide uniformly over the meat surface. In order to eliminate these difficulties a study was made of the suitability of dipping bacon in aqueous solutions of sodium hydroxide.

The material consisted of one freshly-cured Wiltshire back divided into 10 approximately equal portions. Two pieces selected at random were dipped for 10 min. in sodium hydroxide solutions of the following concentrations: 5, 10, 20, 30, and 40%. Each sample was drained for approximately one hour, then wrapped in waxed paper, and overwrapped with brown. Organoleptic examination of the bacon was made after 15 and 30 days' storage at 15.6° C.

The results are shown in Table VII. While retarding the growth of bacteria and moulds, sodium hydroxide failed to prevent spoilage of the bacon. This is in contrast to the rather favourable results previously obtained with sodium

TABLE VII

RESULTS OF ORGANOLEPTIC EXAMINATION OF WILTSHIRE BACON DIPPED IN SOLUTIONS OF SODIUM HYDROXIDE AND STORED AT 15.6°C.

Conc. NaOH, %	Days in storage	Sample No.	Lean				Fat	
			Colour	Odour	Slime	Mould	Colour	Odour
5	15	1	Sl. grey	Sl. off	Moist	Slight	Sl. yellow	Sl. off
		2	Sl. grey	Sl. off	Moist	Slight	Sl. yellow	Sl. off
	30	1	Good	Bad	Heavy	Medium	Sl. yellow	Sl. off
		2	Good	Bad	Heavy	Medium	Sl. yellow	Sl. off
10	15	3	Sl. grey	Sl. off	Sl. moist	None	Sl. yellow	Sl. off
		4	Sl. grey	Sl. off	Very moist	None	Sl. yellow	Sl. off
	30	3	Good	Bad	Heavy	None	Sl. yellow	Sl. off
		4	Dark	Bad	Heavy	None	Sl. yellow	Sl. off
20	15	5	Sl. dark	Sl. off	Very moist	None	Sl. yellow	Sl. off
		6	Sl. dark	Sl. off	Moist	None	Sl. yellow	Sl. off
	30	5	Good	Off	Slight	None	Sl. yellow	Sl. off
		6	Good	Off	Medium	V. slight	Sl. yellow	Sl. off
30	15	7	Sl. dark	Sl. off	Moist	None	Sl. yellow	Sl. off
		8	Sl. dark	Sl. off	Moist	None	Sl. yellow	Sl. off
	30	7	Good	Bad	Moist	V. slight	Sl. yellow	Sl. off
		8	Good	Off	Medium	None	Sl. yellow	Sl. off
40	15	9	Sl. dark	Sl. off	Moist	None	Sl. yellow	Sl. off
		10	Sl. dark	Sl. off	Moist	None	Sl. yellow	Sl. off
		9	Sl. dark	Off	Moist	None	Sl. yellow	Sl. off
		10	Sl. dark	Bad	Moist	None	Sl. yellow	Sl. off

hydroxide applied in the solid form (7). It would appear that sodium hydroxide is more effective as a preservative for bacon when applied as a solid rather than as a solution.

Desiccation

The results of previous investigations have shown that smoking improves the keeping quality of bacon (4, 8, 9). It was considered that preservation might be further enhanced by partial desiccation of the bacon subsequent to smoking.

Materials and Procedure

The material consisted of 12 Wiltshire-cured backs taken from different hogs. After washing and brushing with water, the backs were hung in a small experimental smoke house, and the surface dried for two hours at a temperature of 32.1°C. (90°F.) and an air flow of approximately 500 cu. ft. per min. The air temperature was then raised to 40.6°C. (105°F.) and the backs given a relatively heavy smoke for six hours, after which drying alone was continued.

One back, selected at random, was removed for study and weighed after the following periods (from the initiation of the experiment): 8, 23, and 38 hr.; 4, 7, 8, 9, 12, 15, 17, 21, and 29 days. After removal from the house, the

front end of the back was trimmed and a slice removed for determination of the moisture content of the lean (6). The remainder of the back was wrapped in three layers of waxed paper, overwrapped with brown, and stored at 15.6° C. Organoleptic examinations of the backs and peroxide oxygen determinations on the fat (3) were made after storage for 30 and 60 days, and deep-meat bacterial counts (6) after 30 days.

Results

Data on the loss in weight, moisture content, and deep-meat bacterial counts of the bacon, and the peroxide oxygen content of the fat are given in Table VIII. Desiccation of the backs occurred very slowly, since even after treat-

TABLE VIII

EFFECT OF SMOKING AND PARTIAL DESICCATION ON THE PEROXIDE OXYGEN CONTENT AND DEEP-MEAT BACTERIAL GROWTH IN BACON STORED AT 15.6° C.

Time of treatment	Loss in weight of backs, %	Moisture content of lean meat, %	Peroxide oxygen content, ml. 0.002N Na ₂ S ₂ O ₃		Deep-meat bacterial count after 30 days' storage, log ₁₀ no. per gm.	
			30 days' storage	60 days' storage	Large colonies	Pin-point colonies
Hr.						
8	2.4	67.7	1.2	6.1	8.77	—
23	5.4	68.7	5.0	10.3	6.19	7.33
38	7.1	64.7	13.5	11.0	6.27	0
Days						
4	10.1	64.6	1.8	5.5	8.49	0
7	19.6	60.4	5.9	4.1	4.38	0
8	18.8	55.9	5.1	5.3	3.67	0
9	22.8	58.1	6.6	5.0	5.55	0
12	24.6	56.2	5.3	2.5	0	0
15	30.5	50.9	7.7	1.5	0	0
17	37.3	49.8	9.9	11.3	3.05	0
21	42.4	35.7	10.3	12.9	3.72	0
29	53.6	27.5	22.2	4.9	2.72	0

ment for 29 days the meat still contained 27% moisture. It should also be noted that the values for loss in weight include not only moisture but fat rendered from the backs, especially at the longer drying periods. The peroxide oxygen values of the fat were usually low and somewhat variable, reflecting the antioxidant effect of smoking. Drying to moisture contents of 55 to 60% and lower markedly decreased the deep-meat bacterial counts. The absence of pin-point colonies at the lower moisture levels is considered to be of significance. In previous experiments on the preservation of bacon by hard curing, the presence and development of this type of micro-organism in bacon appeared to be related to the development of stale and off-odours (6).

Results for the organoleptic examination of the backs after storage for 30 and 60 days at 15.6° C. are given in Table IX. Spoilage in the lean meat and slime formation on the surface was prevented by drying to a moisture

TABLE IX
ORGANOLEPTIC EXAMINATION OF SMOKED AND DRIED BACON AFTER STORAGE AT 15.6° C.

Time of treatment	Storage period, days	Lean				Fat, odour	Remarks
		Colour	Odour	Slime	Mould		
Hr.	8	—	Sl. off	—	Heavy	Good	—
	60	Good	Bad	Heavy	Heavy	—	Meat moist and firm
23	30	—	Sl. off	—	Heavy	—	—
	60	Good	Bad	Heavy	Heavy	—	Meat firm
38	30	—	Sl. off	—	Heavy	Good	—
	60	Good	Bad	Heavy	Heavy	—	Meat firm
Days	4	30	Good	Good	Heavy	Good	—
	60	Good	Good	Medium	Heavy	Good	Meat firm
7	30	Good	Excellent	—	Heavy	—	—
	60	Good	Good	—	Heavy	—	Meat slightly separated from fat
8	30	Sl. grey	Good	—	Heavy	—	—
	60	Sl. grey	Good	—	Heavy	—	Meat firm
9	30	Sl. grey	Good	—	Heavy	—	—
	60	Sl. grey	Good	—	Heavy	Good	Meat firm
12	30	Medium grey	Good	—	Medium	—	—
	60	Grey	Good	None	Heavy	Good	Meat cracked and readily separated from fat
15	30	Good	Good	—	Medium	—	—
	60	Dark	Fair	—	Heavy	Good	Meat soft, cracked and partially separated from fat
17	30	Good	Good	—	Slight	—	—
	60	—	Good	None	Slight	Good	Meat cracked and separated from fat
21	30	Brown	Good	None	None	—	—
	60	Brown	Good	None	None	Good	Meat very dry and cracked
29	30	—	—	—	—	—	—
	60	Dark	Good	None	None	Good	Meat very hard, dry and cracked

content of about 55 to 60% and lower. However, a heavy growth of moulds occurred on all backs with moisture contents greater than about 35%. The presence of so much mould was undoubtedly due in large part to the smoking treatment and could possibly be controlled by the application of a mouldicide, such as dimethylol urea. It would appear that a combination of smoking and partial drying, as described here, was comparatively effective in preserving Wiltshire bacon. It is probable that the drying period could be reduced to a more practical value by a suitable selection of drying conditions.

Acknowledgment

The authors wish to thank Dr. N. E. Gibbons, Bacteriologist, National Research Laboratories, for making the bacteriological determinations in the investigation on drying of bacon.

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CANADIAN WILTSHIRE BACON

XXVII. EFFECT OF METHOD OF THAWING FROZEN PORK ON BACON QUALITY¹

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Abstract

Frozen Wiltshire sides were defrosted at temperatures of 4.4°, 12.6°, and 21.0° C. (40°, 55°, and 70° F.) in water; curing pickle; 5, 15, and 30% brines; and in air at high and low relative humidity. Differences due to defrosting procedure were determined by measurement of the thawing period, changes in weight, content of moisture and curing salts, surface bacterial growth, peroxide oxygen formation in the fat, and colour quality and brightness of the lean meat. The effect of method of thawing on keeping quality during storage at -1.1° C. (30° F.) was also studied.

While significant differences were observed between individual thawing treatments in the various criteria employed, few consistent trends could be distinguished between the three temperatures and the four types of media. However, in general it appeared that the more suitable procedures were those that effected thawing within a reasonable period of time. Unduly prolonged exposure to any of the conditions was usually undesirable.

Introduction

Under Canadian conditions it is usually necessary in periods of low hog production to use appreciable quantities of frozen, stored pork for the manufacture of Wiltshire bacon. Suitable conditions for frozen storage have been described previously (1, 2, 5). From two of these studies (1, 5), preliminary data were obtained on the relative suitability of thawing pork in air, water, curing pickle, and a saturated solution of sodium chloride. It was observed that thawing in pickle or brine gave pork a darker colour and more stable fat. The present paper describes a more extensive investigation on the effect of temperature, and the nature and concentration of the thawing medium on surface bacterial growth, colour of the lean meat, and development of rancidity in the fat of pork after conversion to bacon.

Material and Procedure

The right and left sides of 23 hogs were cooled and butchered according to regular commercial practice. After being wrapped with waxed paper and inserted in heavy paper bags, the sides were placed in a sharp-freezer at -26.1° C. (-15° F.) for 24 hr. and subsequently stored at -17.7° C. (0° F.). Two sides, selected at random, were defrosted at temperatures of

¹ Manuscript received April 25, 1945.

Contribution from the Division of Applied Biology, National Research Laboratories, Ottawa. Issued as Paper No. 144 of the Canadian Committee on Food Preservation and as N.R.C. No. 1329.

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4.4°, 12.6°, and 21.0° C. (40°, 55°, and 70° F.), respectively, in each of the following solutions: 5, 15, and 30% sodium chloride; rebuilt Wiltshire cover pickle; water. Two wrapped and two unwrapped sides also were defrosted at the same temperatures in air at an average relative humidity of 48% and, in addition, at 12.6° C. and an average relative humidity of 95%. Systematic differences between sides due to the length of the period of frozen storage prior to defrosting were eliminated by randomizing the order in which the thawing conditions were studied.

Thawing in liquids was carried out in a small wooden tank containing about 60 gal. of the desired solution. By means of a centrifugal pump the liquid was continuously circulated from the bottom of one end of the tank to the top of the opposite end. It was maintained at a constant temperature by passage through a thermostatically controlled heat exchanger. Thawing in air was accomplished by hanging the sides in a small, insulated room provided with suitable controls for temperature and relative humidity.

Temperature changes in each side during thawing were followed by thermocouples inserted to a depth of about 2 in. in each of the following three positions: in the gammon at a point 5 in. from the top edge of the side and 3.5 in. from the femur; in the back opposite the eighth rib and 2 in. from the top edge; and in the fore-end 5.5 in. anterior to the first rib and 5 in. from the top edge of the side. Temperature readings of the sides were made at 15 to 60 min. intervals, depending on the temperature of the thawing, until all three points had reached 1.6° C. (35° F.) or higher.

After removing the sides from the thawing bath or chamber, they were weighed, drained for one day, and reweighed. The sides were pumped with Wiltshire pump pickle by commercial operators, weighed, and cured at 4.4° C. (40° F.) for four days in small tanks containing Wiltshire cover pickle. (The short curing period was employed because of the abnormally large pickle to meat ratio associated with the use of the small tank (6)). After removal from cure, the sides were drained for one day, weighed, and cut up to obtain the long rib-in English style backs.

A sample was removed from the rear of each back for determination of the chloride, nitrate, nitrite, and moisture content of the lean meat (4, 7); and from the front, for measurement of the colour (8, 9). Samples (3 sq. cm.) of surface tissue, removed from each of three approximately equidistant positions along the back, were taken for bacterial counts (3). The backs were then wrapped in waxed paper, overwrapped in brown, and stored at -1.1° C. (30° F.). Bacterial counts were repeated after 30 and 60 days' storage, and colour measurements of the meat and peroxide oxygen determinations on the fat after 30, 60, and 90 days.

Results

Defrosting Time

At any one temperature the time required for defrosting was approximately of the same order of magnitude in all the liquid media studied, and considerably shorter than in air of low relative humidity (Table I). Wrapping the

TABLE I

EFFECT OF TEMPERATURE AND MEDIUM OF THAWING ON THE PERIOD REQUIRED TO THAW FROZEN WILTSHIRE SIDES

Thawing medium	Temperature of thawing medium, °C.	Time ¹ , hr.	Temperature ¹ (°C.) of:		
			Fore-end	Back	Gammon
Brine 5%	4.4	24.3	3.6	6.3	1.7
	12.6	12.3	3.9	12.5	1.7
	21.0	6.9	9.0	18.6	1.7
15%	4.4	27.2	5.0	5.8	1.7
	12.6	14.8	3.0	13.1	1.7
	21.0	6.8	4.9	15.1	1.7
30%	4.4	22.3	1.9	5.6	1.7
	12.6	10.2	4.6	12.3	1.7
	21.0	5.3	2.2	15.9	1.7
Wiltshire curing pickle	4.4	28.0	2.6	4.5	1.7
	12.6	13.6	5.5	12.8	1.7
	21.0 ²	7.8	11.1	17.0	1.7
Water	4.4	21.1	2.1	3.7	1.7
	12.6	8.2	7.5	11.3	1.7
	21.0	9.3	8.1	19.6	1.7
Air, low R. H. Wrapped	4.4	111.7	2.9	4.2	1.7
	12.6	51.4	4.1	7.2	1.7
	21.0 ²	37.4	12.2	15.6	1.7
Unwrapped	4.4	52.1	2.7	2.8	1.7
	12.6	29.7	6.0	9.7	1.7
	21.0	24.2	15.0	11.8	1.7
Air, high R. H. Wrapped	12.6	47.5	6.3	5.4	1.7
	12.6	13.7	4.7	9.7	1.7

¹ Mean values for two sides.² Values obtained on one side only.

sides materially lengthened the defrosting period. An increase in the relative humidity had little effect on the period required for wrapped sides at the one temperature studied, but reduced the time for unwrapped sides to about that observed for pickle, brine, or water.

With air defrosting at a low relative humidity and with water there was a marked difference between the times required at 4.4° and 12.6° C., and a considerably smaller difference between 12.6° and 21.0° C. In the other media an increase in temperature of 8.2° C. effected a reduction of about one-half in the defrosting period.

Changes in Weight

Mean values for changes in weight of the sides during thawing, pumping, and curing are given in Table II. Sides held in any of the liquids or in air of high relative humidity increased in weight during thawing, while those treated

TABLE II

EFFECT OF TEMPERATURE AND MEDIUM OF THAWING ON CHANGES IN WEIGHT OF WILTSHIRE SIDES DURING THAWING, PUMPING, AND CURING

Thawing medium	Temperature of thawing medium, °C.	Change in weight of sides ¹ , %		
		During thawing	During pumping and curing	During thawing, pumping, and curing
Brine 5%	4.4	3.8	2.6	5.6
	12.6	3.4	3.2	6.3
	21.0	2.6	4.9	6.7
15%	4.4	3.5	2.8	6.3
	12.6	2.6	4.0	6.4
	21.0	2.6	4.4	6.8
30%	4.4	0.8	4.6	5.7
	12.6	0.6	4.7	5.5
	21.0	0.9	3.8	4.3
Wiltshire curing pickle	4.4	0.5	2.7	3.4
	12.6	1.4	4.8	6.1
	21.0	1.1	3.2	6.6
Water	4.4	3.4	6.3	9.2
	12.6	3.0	3.4	6.4
	21.0	2.4	3.6	6.2
Air, low <i>R. H.</i> Wrapped	4.4	-1.2	4.6	3.7
	12.6	-0.8	8.6	7.9
	21.0	-0.6	5.4	4.7
Unwrapped	4.4	-3.4	4.0	2.3
	12.6	-1.5	7.3	6.1
	21.0	-1.9	7.4	5.5
Air, high <i>R. H.</i> Wrapped	12.6	2.0	3.8	5.0
	12.6	0.2	5.4	6.0

¹ Mean values for two sides.

in air of low relative humidity decreased. Increases in weight were greatest and approximately of the same order of magnitude with the use of 5 and 15% brine; water; and, for wrapped sides, air of high relative humidity. For sides defrosted in air, wrapping decreased the losses in weight occurring at the low relative humidity and increased the gains in weight at the high humidity. While mean differences due to defrosting temperatures were for the most part small, the greatest changes in weight were usually associated with a defrosting temperature of 4.4°C. This behaviour is presumably a reflection of temperature on the length of the thawing period.

As was to be expected, all sides increased in weight during pumping and curing. The increase was, in general, greatest for those conditions that caused minimum changes during thawing.

The over-all increases in weight as a result of thawing, pumping, and curing showed no systematic differences within or between the various groups of defrosting conditions. However, when averaged over all thawing media the increase in weight was greatest at 12.6° and least at 4.4° C.

Content of Moisture and Curing Salts

Mean values for the content of moisture, chloride, nitrate, and nitrite in the back bacon after cure are given in Table III. Variations in moisture

TABLE III

EFFECT OF TEMPERATURE AND MEDIUM OF THAWING ON THE CONTENT OF MOISTURE AND CURING SALTS IN WILTSHIRE BACK BACON

Thawing medium	Temperature of thawing medium, ° C.	Component ¹			
		Moisture, %	Sodium chloride, %	Sodium nitrate, %	Sodium nitrite, p.p.m.
Brine 5%	4.4	68.2	5.56	0.13	40
	12.6	70.2	5.52	0.11	33
	21.0	68.5	4.94	0.13	67
15%	4.4	69.2	5.97	0.12	53
	12.6	69.5	4.18	0.10	34
	21.0	66.0	8.04	0.22	97
30%	4.4	68.6	5.38	0.21	53
	12.6	71.0	3.43	0.07	18
	21.0	69.0	5.47	0.08	27
Wiltshire curing pickle	4.4	69.1	5.89	0.16	42
	12.6	69.2	6.54	0.16	51
	21.0	65.1	7.01	0.21	36
Water	4.4	71.2	4.43	0.14	68
	12.6	72.0	5.07	0.11	43
	21.0	69.8	6.01	0.18	33
Air, low R. H. Wrapped	4.4	68.8	4.21	0.18	54
	12.6	67.0	5.62	0.14	72
	21.0	70.1	5.95	0.20	47
Unwrapped	4.4	69.9	4.74	0.16	26
	12.6	71.8	3.38	0.11	53
	21.0	71.8	5.95	0.09	59
Air, high R. H. Wrapped	12.6	70.6	4.04	0.10	40
	12.6	76.0	6.25	0.14	92

¹ Mean values of duplicate determinations for each of two sides.

content showed no systematic trends between the temperatures or thawing media studied. However, the values, as averaged over all temperatures, were greatest for unwrapped sides thawed in water or in air of low relative humidity and least for those held in pickle or in air of high relative humidity.

The nature of the thawing medium had little effect on the content of curing salts, indicating that brine or curing pickle can be employed without giving an undesirably salty product. The highest concentrations of sodium chloride were usually associated with a temperature of 21.0° C. However, with this possible exception there was little to indicate the superiority of any particular defrosting condition.

Surface Bacterial Growth

Surface bacterial counts on the back bacon prior to storage were, on the average, lowest for those sides that had been defrosted in 15 or 30% brine, in curing pickle, or, unwrapped, in air of low relative humidity and a temperature of 21.0° C. (Table IV). Presumably the effect of temperature is, in large part, a reflection of the length of time required for thawing and suggests that

TABLE IV

EFFECT OF TEMPERATURE AND MEDIUM OF THAWING ON SURFACE BACTERIAL GROWTH ON WILTSHIRE BACK BACON DURING SUBSEQUENT STORAGE AT -1.1° C. (30° F.)

Thawing medium	Temperature of thawing medium, ° C.	Log ₁₀ number of organisms per sq. cm. ¹		
		Storage period, days		
		0	30	60
Brine 5%	4.4	6.39	6.93	8.50
	12.6	4.96	6.90	8.55
	21.0	6.36	8.92	8.33
15%	4.4	4.53	8.44	9.09
	12.6	4.28	4.66	6.79
	21.0	5.42	6.72	8.48
30%	4.4	4.32	5.44	7.60
	12.6	4.28	6.26	8.75
	21.0	4.62	7.43	9.12
Wiltshire curing pickle	4.4	5.65	6.68	8.31
	12.6	4.99	9.45	8.89
	21.0	3.46	6.41	7.80
Water	4.4	7.28	8.76	8.93
	12.6	6.20	8.90	8.89
	21.0	4.27	3.98	8.13
Air, low <i>R. H.</i> Wrapped	4.4	5.65	8.79	9.19
	12.6	5.56	8.75	9.08
	21.0	5.21	8.01	9.04
Unwrapped	4.4	4.16	7.50	9.02
	12.6	5.29	9.06	9.20
	21.0	4.59	6.55	7.98
Air, high <i>R. H.</i> Wrapped	12.6	6.48	9.10	8.58
	Unwrapped	12.6	7.73	8.86

¹ Mean values for two sides.

this period should be kept to a minimum. Wrapped sides defrosted in air tended to have higher surface bacterial counts.

Surface bacterial growth occurred during storage at $-1.1^{\circ}\text{C}.$; the rate of increase was greatest during the first 30 days storage, and was relatively greater the lower the initial count. Differences between sides defrosted by the various procedures were small after storage for 60 days and were of little statistical significance.

Peroxide Oxygen Formation in the Fat

The peroxide oxygen values of the back bacon fat were variable in behaviour with respect both to the defrosting condition and the subsequent storage period at $-1.1^{\circ}\text{C}.$ (Table V). However, lower peroxide oxygen values were obtained by thawing the unwrapped sides in liquid media, at the

TABLE V

EFFECT OF TEMPERATURE AND MEDIUM OF THAWING ON PEROXIDE OXYGEN FORMATION IN THE FAT OF WILTSHIRE BACK BACON DURING SUBSEQUENT STORAGE AT $-1.1^{\circ}\text{C}.$ ($30^{\circ}\text{F}.$)

Thawing medium	Temperature of thawing medium, $^{\circ}\text{C}.$	Peroxide oxygen content ¹ , ml. 0.002 N $\text{Na}_2\text{S}_2\text{O}_3$		
		Storage period, days		
		30	60	90
Brine 5%	4.4	6.0	10.2	19.9
	12.6	14.7	14.6	11.1
	21.0	21.0	16.6	24.0
15%	4.4	6.9	12.2	7.8
	12.6	20.1	27.0	10.1
	21.0	5.6	13.8	13.3
30%	4.4	30.1	33.2	16.7
	12.6	22.0	12.0	10.4
	21.0	5.2	10.0	10.2
Wiltshire curing pickle	4.4	7.9	6.5	8.8
	12.6	10.4	10.3	13.2
	21.0	40.9	26.3	25.5
Water	4.4	6.1	4.7	9.1
	12.6	21.7	19.5	13.1
	21.0	26.1	22.8	23.8
Air, low $R. H.$ Wrapped	4.4	30.7	27.3	16.2
	12.6	7.8	5.7	8.3
	21.0	4.9	8.3	8.6
Unwrapped	4.4	6.6	10.8	8.2
	12.6	3.8	7.1	3.4
	21.0	8.2	15.5	9.7
Air, high $R. H.$ Wrapped	12.6	11.7	4.0	10.9
	12.6	16.9	18.0	14.3

¹ Mean values of duplicate determinations for each of two sides.

lower temperatures, and wrapped sides in air of low humidity, at the higher temperatures; these conditions would, therefore, seem to be more suitable.

In a previous investigation (5), in which small cuts of pork were used, it was observed that defrosting in saturated brine or pickle at 4.5° C. usually yielded a product with slightly less peroxide oxygen than did thawing in air at 10.0° C. or water at 38.0° C. The present results are in substantial agreement with these previous conclusions when consideration is given to the differences in experimental conditions employed.

Colour Brightness and Quality

Mean values for colour brightness are given in Table VI. Neither temperature nor thawing medium had significant effect on colour brightness. A general and statistically significant increase in brightness occurred during storage at -1.1° C.

TABLE VI

EFFECT OF TEMPERATURE AND MEDIUM OF THAWING ON CHANGES IN COLOUR BRIGHTNESS OF WILTSHIRE BACK BACON DURING SUBSEQUENT STORAGE AT -1.1° C. (30° F.)

Thawing medium	Temperature of thawing medium, ° C.	Total scatter ¹ , %	
		Storage period, days	
		0	90
Brine 5%	4.4	11.8	13.0
	12.6	10.3	11.8
	21.0	10.9	12.2
15%	4.4	11.7	13.3
	12.6	11.4	11.5
	21.0	12.9	13.6
30%	4.4	11.4	11.8
	12.6	14.5	12.4
	21.0	11.5	12.0
Wiltshire curing pickle	4.4	11.9	12.3
	12.6	11.2	12.7
	21.0	11.0	11.9
Water	4.4	10.5	12.7
	12.6	10.4	12.2
	21.0	12.4	12.6
Air, low R. H. Wrapped	4.4	12.5	13.4
	12.6	12.0	12.1
	21.0	10.4	12.4
Unwrapped	4.4	10.4	11.3
	12.6	13.9	13.9
	21.0	10.7	12.6
Air, high R. H. Wrapped	12.6	12.8	12.0
	12.6	12.7	13.4

¹ Mean values of duplicate determinations for each of two sides.

While data were obtained for nine colour bands (9), statistical analyses indicated that the method of defrosting had little significant effect on colour quality. Accordingly, the data have been grouped for more ready comparison of over-all changes in the blue, green, and red spectral regions (Table VII). During the storage at -1.1°C ., the light scattered in the blue region decreased; green scatter increased; while red scatter decreased after 30 days and subsequently increased.

TABLE VII

EFFECT OF TEMPERATURE AND MEDIUM OF THAWING ON COLOUR QUALITY OF WILTSHIRE BACK BACON DURING SUBSEQUENT STORAGE AT -1.1°C . (30°F .)

Thawing medium	Temperature of thawing medium, $^{\circ}\text{C}$	Mean scatter ¹ , %					
		Blue, 3850-5250 Å		Green, 5250-5840 Å		Red, 5840-6440 Å	
		0 days	90 days	0 days	90 days	0 days	90 days
Brine 5%	4.4	48.8	38.6	25.5	28.7	25.7	32.7
	12.6	45.7	43.1	27.7	28.1	26.6	29.0
	21.0	46.2	43.2	26.4	29.1	27.6	28.1
15%	4.4	47.7	39.0	25.8	28.6	26.6	32.4
	12.6	47.6	46.8	27.4	26.7	25.1	27.1
	21.0	48.4	40.9	26.3	28.1	25.5	31.1
30%	4.4	44.3	45.2	26.8	28.1	28.9	26.8
	12.6	41.1	44.0	29.1	28.8	30.0	27.3
	21.0	46.5	42.0	25.7	29.2	27.9	28.9
Wiltshire curing pickle	4.4	47.8	41.9	26.0	27.5	26.2	30.7
	12.6	47.6	41.0	26.3	30.3	26.5	28.8
	21.0	45.6	47.0	27.3	27.0	27.2	26.2
Water	4.4	47.1	41.6	27.0	28.6	25.9	29.9
	12.6	47.7	41.3	25.3	29.2	27.7	29.7
	21.0	48.4	46.4	26.9	27.1	24.7	26.6
Air, low R. H. Wrapped	4.4	44.2	44.4	27.7	28.9	28.2	26.9
	12.6	45.9	42.7	27.7	28.7	26.6	28.7
	21.0	45.9	40.3	26.7	28.1	27.3	31.6
Unwrapped	4.4	48.1	42.5	25.6	28.4	26.5	29.7
	12.6	46.8	39.3	26.8	30.7	26.6	32.9
	21.0	46.3	40.3	26.3	27.3	27.6	32.5
Air, high R. H. Wrapped	12.6	46.7	42.2	26.7	28.6	26.6	28.9
	12.6	48.0	42.7	26.7	28.6	25.5	28.4

¹ Mean values of duplicate determinations for each of two sides.

Conclusions

Under the conditions employed in this study no one thawing medium appeared to be markedly superior to the others. In air, wrapping prolonged, but a high relative humidity accelerated, the defrosting. Thawing in all the liquid media occurred at approximately the same rate. Increases in weight during thawing were greater in 5 than 30% brine or in curing pickle. The

smallest defrosting gains or losses were associated with a temperature of 21.0° C. After cure there was little difference in weights between sides defrosted by the various procedures. However, there was some indication that, on the average, a temperature of 12.6° C. and water or brine of 5 or 15% concentration favoured maximal increases in weight. While the various defrosting media had no marked effect on the content of moisture and curing salts, a higher thawing temperature tended to give a somewhat saltier product.

The surface bacterial counts tended to be somewhat lower when the sides were defrosted at 21.0° C. in water, curing pickle, or the more concentrated brines. The highest mean count was obtained on sides defrosted in water at 4.4° C. In air either wrapping or a high relative humidity enhanced surface bacterial growth. Defrosting conditions likewise had little specific effect on peroxide oxygen formation in the fat. In general a low temperature was more suitable for the liquid media, and a high temperature for air at low relative humidity with wrapped sides. The defrosting methods studied appeared to have no systematic influence on either colour, quality, or brightness.

While the somewhat indefinite character of the results precludes the possibility of definite conclusions, there is some indication that liquid media or air at high humidity may possibly be superior to dry air and that a defrosting temperature of 12.6° C. merits commercial trial.

Acknowledgments

The kind co-operation of Mr. W. Hodder, Superintendent of Canada Packers Ltd., Hull, Que., which made the investigation possible, is gratefully acknowledged. The authors also wish to thank Miss W. Price for technical assistance and Mr. D. B. W. Reid for making the statistical computations.

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SEPARATION OF STARCH AND GLUTEN

III. A RAPID METHOD OF SEPARATION FROM WHEAT FLOUR¹

BY A. L. SHEWFELT² AND G. A. ADAMS²

Abstract

Patent flour was mixed with sufficient water to form a smooth coherent dough, which was subsequently broken up into curds by agitating with additional water. Primary separation of the starch suspension and crude gluten was effected by passing the slurry over an inclined gyrating screen of about 150 mesh. Starch was recovered from the suspension by means of a basket-type centrifuge and dried. More than 90% of the starch containing less than 0.5% of protein was recovered. Substantially complete recovery of the gluten having a starch content of about 20% (dry basis) was realized. The crude gluten could be readily refined by shredding, washing, and rescreening. When dry, it contained 70 to 75% of protein and 10 to 12% of starch.

A study of the effects of the processing factors has shown the following permissible ranges: amount of water for dough-mixing, equivalent to 80 to 85% of the weight of flour; temperature of the water for mixing, 25° to 30° C.; time of dough-mixing, 15 to 20 min.; time of dough agitation in water, 1 to 4 min. A portion of the dough-mixing time could be replaced by a dough-conditioning period. The most critical factors were the flour-water ratio and the time of dough-agitation.

The method effected a rapid and efficient separation of the starch and gluten fractions and should be adaptable to continuous large-scale operation.

Introduction

An earlier communication from these laboratories reviewed several methods for preparing starch and gluten from wheat (1). Recently, renewed interest in this field has brought out additional improved methods of production. Slotter and Langford (8) have reported the successful application to wheat of wet milling principles used in corn starch manufacture. In a process described by Dimler *et al.* (5), starch has been prepared from various cereal flours by dissolving the gluten in dilute alkali. The main features of the method described herein have been reported previously (7), and are similar to those of a process reported at about the same time by Hilbert *et al.* (6). This communication describes the results of detailed studies on the effects of various factors involved in the separation, and their bearing on the practical operation of the process.

Materials and Methods

Separation Procedure

A soft coherent dough was prepared by mixing 3 kgm. of commercial patent flour with about 80% of its weight of tap water (pH 7.0 to 8.0) at slow speed in a laboratory type Hobart dough-mixer. The dough was transferred with 10 kgm. of water to an agitation vessel equipped with a reversing paddle,

¹ Manuscript received in original form April 26, 1945, and as revised July 30, 1945.

Contribution from the Division of Applied Biology, National Research Laboratories, Ottawa. Issued as Paper No. 29 on the Industrial Utilization of Wastes and Surpluses, and as N.R.C. No. 1332.

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where it was broken into curd-like fragments about 1 cm. in diameter from which the starch could be readily washed. The heterogeneous mixture was pumped by a Jabsco rubber impeller pump* to a Rotex No. 11 screener** equipped with a screen of about 150 mesh. The starch milk passed through the screen while the crude gluten was washed with a spray of water and carried over the end. The screen sloped at a 4° angle and was operated at a speed of 530 gyrations per minute.

Gluten Recovery

The crude gluten was collected on a coarse sieve at the lower end of the gyrating screen. When a 165 mesh silk screen was used in the separation (as in Series I and II) sufficient starch milk was carried over with the gluten to warrant rinsing the latter with 3 to 4 kgm. of water and recycling the liquid to the screen. In Series III the silk screen was replaced by a 150 mesh phosphor bronze screen, which eliminated the necessity of rinsing and recycling.

The wet, crude gluten (about 1000 gm.) from the screening operation was extruded in the form of curdlike shreds through a screw-type food chopper. It was then washed with 4 kgm. of water for two to three minutes in the agitation vessel, rescreened, and drum dried at 70 lb. steam pressure.

Starch Recovery

A basket centrifuge fitted with three layers of filter cloth was used to recover wet starch from aliquots of the starch suspension. The wet starch was scraped from the filter cloth into shallow trays and dried in a current of air at 35° C. for 20 hr. Its moisture content was then brought to equilibrium in air.

Analytical Procedures

Starch content of the air-dry flour, starch, and gluten samples was determined according to the methods of Clendenning (3, 4). The nitrogen content of these flour and starch samples was determined by the Kjeldahl method and converted to corresponding protein values by the factor 5.7. Moisture content of the flour and drum-dry gluten was determined by drying at 130° C. for two hours according to the A.A.C.C. electric air-oven method (2). Gluten content of the flour was determined by the A.A.C.C. hand-washing method (2), and formed a basis for calculating gluten recoveries in the various separations. Analyses of the flours used in the various experiments are given in Table I.

Experimental

The experimental investigation included three separate series of experiments. The first related to factors affecting dough preparation and included only those combinations of factors amenable to successful treatment. The agitation or slurring phase in each experiment was carried to the point where the gluten particles were about 1 cm. in diameter and could be pumped readily

* Obtained from Enterprise Agencies Ltd., Montreal, P.Q.

** Obtained from Orville Simpson Co., Ltd., Cincinnati, Ohio.

TABLE I
ANALYSIS OF FLOURS USED IN THE VARIOUS EXPERIMENTS

Component	Series I and II		Series III	
	%	Wt. in 3 kgm. flour, gm.	%	Wt. in 3 kgm. flour, gm.
Starch	67.6	2030	67.1	2013
Protein	11.8	354	13.0	390
Moisture	12.4	330	15.0	390
Gluten	12.0	360	12.4	372

through a $\frac{3}{4}$ in. hose to the screen. The time of agitation varied from one-half to three minutes. A number of experiments in which part of the dough-mixing time was replaced by a dough-standing or conditioning period was included. A later study was made of the breaking up of the dough prior to screening using the conditions listed in Table V. These led to a factorial treatment of the experimental conditions involved in the separation (see Table VI). The 165 mesh silk screen employed in the earlier studies was replaced in this series by a 150 mesh phosphor bronze screen, since adhering gluten could be entirely removed from the latter by a dilute ammonia wash.

The effects of the various factors were determined from the yields and purity of the recovered starch and gluten fractions.

Results

FACTORS AFFECTING DOUGH PREPARATION (SERIES I)

Data on the effects of temperature and volume of mixing water, and of time of mixing are given in Table II. In most experiments, the gluten recovery was substantially complete. In others, Nos. 13, 17, 20, and 30, the recoveries were lower owing to the unfavourable dough-preparation conditions used.

Experiments in which 2100 ml. of mixing water was used were not satisfactory owing to the high starch content of the gluten. The starch content was much lower when 2550 ml. was used, but the dough was friable and allowed more gluten to pass through the screen. The protein content of the starch was therefore higher.

Data from these experiments in which 2250 and 2400 ml. were used (with the exception of Nos. 13, 20, 28, and 29) were subjected to a statistical analysis of variance. Mean squares for starch and gluten recoveries and purities are presented in Table III.

The data in Table III show that starch recoveries for the 10 and 15 min. mixing times were both 87% of the total starch present, while that for the 25 min. time was 90%. The 2400 ml. volume favoured a higher starch recovery than the 2250 ml. volume although the difference was not statistically significant.

TABLE II

SERIES Ia. EFFECTS OF TEMPERATURE AND VOLUME OF MIXING WATER AND TIME OF MIXING ON THE SEPARATION OF STARCH AND GLUTEN

Expt. No.	Experimental factors			Results, %				
	Temp. of water, ° C.	Volume of mixing water, ml.	Time of mixing, min.	Starch recovery	Protein content of starch	Gluten recovery	Starch content of gluten	Total starch in gluten fraction
1	20	2100	5	80	0.50	84	43	11.4
2			10	81	.48	100	42	12.7
3			15	85	.47	98	43	13.7
4		2250	10	83	.44	93	33	8.2
5			15	84	.40	95	32	7.8
6			25	89	.40	99	32	8.3
7		2400	10	86	.52	85	25	5.1
8			15	88	.44	88	27	5.8
9			25	89	.44	100	30	7.7
10	25	2100	5	80	.47	103	41	12.7
11			10	78	.44	103	33	9.4
12			15	86	.40	105	37	11.7
13		2250	5	84	.50	76	33	6.7
14			10	85	.46	94	38	10.1
15			15	88	.48	96	32	10.5
16		2400	25	91	.44	101	28	7.0
17			10	87	.47	84	20	3.6
18			15	88	.46	99	23	5.4
19	30	2250	25	89	.44	100	23	5.4
20			5	92	.46	88	31	6.9
21			10	89	.49	94	33	8.0
22		2400	15	86	.48	102	32	8.6
23			25	88	.51	101	33	8.9
24			10	90	.45	98	26	6.1
25		2550	15	88	.43	100	29	7.2
26			25	93	.41	96	25	5.7
27			25	91	.58	94	18	3.7
28	35	2250	10	80	.48	95	34	9.7
29		2400	15	86	.40	95	26	5.9
30		2550	25	91	.54	88	24	5.1

TABLE III

ANALYSIS OF VARIANCE OF THE EFFECTS OF TEMPERATURE AND VOLUME OF MIXING WATER AND TIME OF MIXING ON STARCH AND GLUTEN SEPARATION

Source of variation	Degrees of freedom	Mean squares			
		Starch recovery	Protein content of starch	Gluten recovery	Starch content of gluten
Temperature	2	9.5	8.1	40.2	11.5
Volume	1	12.5	0.9	34.7	235.0**
Mixing time	2	18.1*	16.1	103.2*	1.0
Temp. × vol.	2	3.1	50.6**	5.1	13.5
Error	10	4.6	4.7	17.1	7.8

* Exceeds 5% level of significance.

** Exceeds 1% level of significance.

The volume of mixing water had a significant effect on the starch content of the gluten, the average being 25% with 2400 ml. and 32% with 2250 ml.

The analysis of variance for protein content of starch showed a highly significant interaction between temperature and volume. At 20° C. the average protein level was 0.41% with 2250 ml. and 0.47% with 2400 ml. At 30° C. the levels were 0.49 and 0.43%, respectively. The longer mixing times of 15 and 25 min. tended to decrease the protein content of the starch, though differences were not significant.

Dough Conditioning

The data presented in Table IV show that a short mixing time followed by conditioning gave as effective separations as did continuous mixing for the same total length of time. Increasing the conditioning period tended to result in higher starch recovery, but it also increased the protein content of the starch. It must be noted also that prolonged conditioning required increased agitation time for the preparation of satisfactory slurries, with the result that the conditioning effects were partially masked.

TABLE IV

SERIES Ib. EFFECT OF DOUGH CONDITIONING IN AIR AND IN WATER AT 30° C.

Expt. No.	Experimental factors					Results, %			
	Conditioning time, min.	Conditioning medium	Volume of mixing water, ml.	Mixing time, min.	Agitation time, min.	Starch recovery	Protein content of starch	Starch content of gluten	Total starch in gluten
1	5	Air	2400	5	1.0	84	0.43	30	7.9
2	10	Air			1.5	83	.40	31	8.4
3	15	Air			1.5	86	.50	30	7.8
4	5	Air		10	1.25	87	.41	28	6.9
5	10	Air			1.5	87	.48	28	7.1
6	15	Air			2.0	90	.46	24	5.7
7	5	Air	2500	15	1.5	86	.44	29	7.5
8	10	Air			2.0	86	.49	27	6.8
9	10	Air		10	1.0	89	.46	26	6.4
10	20	Air			1.25	92	.44	23	5.5
11	30	Air			1.25	93	.51	25	6.3
12	10	Air		15	1.5	93	.48	27	6.7
13	20	Air			1.75	92	.55	22	5.5
14	30	Air			1.5	93	.52	22	5.4
15	5	Water	2400	10	1.25	86	.51	24	5.8
16	10	Water			1.0	87	.50	26	6.5
17	15	Water			1.25	92	.57	30	7.4
18	10	Water	2550	15	1.0	92	.47	24	5.9
19	20	Water			1.0	93	.56	22	5.2
20	30	Water			1.5	93	.52	21	4.9

The protein content of the starch was higher for water than for air-conditioned doughs, while the starch content of the glutens did not differ substantially. Water-conditioning has possible advantages from the standpoint of dough conveyance in continuous commercial operation.

AGITATION (SERIES II)

Table V shows that when 2400 ml. of mixing water was used, increasing the agitation time from 1.5 to 3.0 min. increased the starch recovery from 82 to 96% and decreased the starch content of the gluten from 32 to 23%. Gluten

TABLE V

SERIES II. EFFECT OF AGITATION TIME ON THE SEPARATION OF STARCH AND GLUTEN USING DOUGHS MIXED FOR 15 MIN. AT 30° C.

Expt. No.	Experimental factors			Results, %			
	Agitation time, min.	Volume of mixing water, ml.	Conditioning time, min.	Starch recovery	Protein content of starch	Starch content of gluten	Total starch in gluten
1	1.5	2400	5	82	0.37	32	9.5
2	2.25			89	.45	25	6.7
3	3.0			96	.43	23	5.9
4	1.25	2550	10	87	.46	27	6.7
5	2.0			95	.50	19	4.2
6	2.75			96	.52	17	3.4

recovery was substantially complete and the protein content of the starch did not exceed 0.45%. With 2550 ml., extending the agitation time from 1.25 to 2.75 min. increased the starch recovery from 87 to 96% and lowered the starch content of the gluten from 27 to 17%. The longer agitation time, however, resulted in a protein level of 0.52% in the starch, indicating that the time of agitation must be limited for optimum separation.

FACTORIAL STUDY OF FACTORS AFFECTING THE SEPARATION (SERIES III)

A tabulation of the experimental factors and results appear in Table VI and a summary of the analysis of variance for these data is given in Table VII.

Starch Recovery

Experimental factors significantly affecting starch recovery included agitation time, conditioning time, and volume of mixing water. The optimum agitation time was three minutes. Increasing the conditioning time diminished the starch recovery. It will be recalled that in Series I, in which agitation time was not fixed, the opposite effect was observed. As in Series I, starch recovery was affected by volume of mixing water, the averages being 87% with 2550 ml. and 77% with 2400 ml. In general, the 15 min. mixing time tended to give higher starch recoveries, although there was no significant difference between 15 and 25 min.

Table VII shows two significant interactions. That between agitation and conditioning time was such that at the shorter agitation times conditioning the dough had a depressing effect on starch recovery while at the four minute period this effect was no longer apparent. The interaction between agitation time and volume of mixing water showed that starch recovery was higher

TABLE VI

SERIES III. DATA ON THE FACTORIAL TREATMENT OF FACTORS AFFECTING THE SEPARATION OF STARCH AND GLUTEN FROM DOUGHS MIXED AT 30° C.

Expt. No.	Experimental factors				Results, %				
	Volume of mixing water, ml.	Mixing time, min.	Conditioning time, min.	Agitation time, min.	Starch recovery	Protein content of starch	Gluten recovery	Starch content of gluten	Total starch in gluten
1	2400	15	0	1	80	0.52	98	42	13.5
2				2	84	.52	102	34	10.2
3				3	83	.59	95	31	8.1
4				4	87	.48	98	24	5.9
5		10	10	1	54	.45	100	64	38.3
6				2	83	.44	105	31	9.2
7				3	83	.41	101	26	7.0
8				4	83	.42	96	26	6.2
9		20	20	1	58	.46	97	55	30.5
10				2	79	.50	100	43	15.2
11				3	82	.50	98	29	7.7
12				4	86	.41	94	25	5.8
13		25	0	1	72	.52	103	48	18.7
14				2	82	.46	98	30	8.2
15				3	89	.54	100	27	7.2
16				4	83	.56	95	27	6.8
17		10	10	1	63	.50	100	57	26.7
18				2	77	.44	98	44	15.0
19				3	84	.53	100	29	7.9
20				4	85	.42	95	24	5.8
21		20	20	1	49	.54	101	66	45.8
22				2	66	.54	98	59	28.4
23				3	85	.48	100	30	8.0
24				4	79	.42	94	30	7.6
25	2550	15	0	1	84	.46	98	35	10.0
26				2	90	.53	98	22	5.4
27				3	91	.62	96	23	5.5
28				4	90	.50	99	20	4.5
29		10	10	1	81	.44	100	38	12.1
30				2	85	.68	96	25	6.0
31				3	85	.60	95	22	4.8
32				4	91	.55	100	18	4.2
33		20	20	1	80	.55	105	38	12.3
34				2	83	.53	96	25	6.1
35				3	88	.50	101	20	4.7
36				4	87	.55	97	19	4.2
37		25	0	1	89	.44	102	29	7.8
38				2	93	.51	102	22	5.2
39				3	90	.43	98	21	4.8
40				4	88	.59	94	18	3.9
41		10	10	1	74	.54	105	42	14.9
42				2	86	.68	101	22	5.5
43				3	90	.46	100	20	4.8
44				4	88	.42	104	19	4.5
45		20	20	1	67	.51	99	55	24.2
46				2	91	.47	102	25	6.4
47				3	91	.50	99	20	4.8
48				4	93	.50	100	19	4.4

TABLE VII

ANALYSIS OF VARIANCE OF DATA FROM SERIES III

Source of variation	Degrees of freedom	Mean square			
		Starch recovery	Protein content of starch	Gluten recovery	Starch content of gluten
Agitation time	3	674.8**	39.4	27.3*	1501.1**
Mixing time	1	11.0	9.2	11.4	46.2
Conditioning time	2	208.3**	18.8	6.8	166.8**
Volume	1	999.2**	172.5*	9.2	1683.9**
AT \times CT	6	65.9**	44.1	2.8	69.9**
AT \times Vol.	3	84.9**	58.1	14.6	78.3**
MT \times Vol.	1	22.7	105.0	14.8	23.7
CT \times Vol.	2	27.4	119.2*	5.6	25.1
Error	28	17.6	30.6	7.2	21.5

* Exceeds 5% level of significance.

** Exceeds 1% level of significance.

with the larger amounts of mixing water at all four agitation times. The differences, however, were much more marked at the one minute agitation time, than at the longer periods. With 2400 ml., the starch recovery levelled off at the three minute agitation time, while with 2550 ml. it became constant after two minutes.

Protein Content of Starch

Volume of mixing water was the only primary factor significantly affecting protein content of the starch. The mean protein content was 0.48% with 2400 ml. and 0.52% with 2550 ml. In general the differences were small and erratic, and in commercial practice a dilute alkali wash would probably be required to obtain a product of uniformly low protein content.

Gluten Recovery

In most experiments complete recovery of gluten was realized. Agitation time was the only factor that affected recovery significantly. Mean recoveries for one, two, three, and four minutes were 100.7, 99.7, 98.6, and 97.2%, respectively. Since the necessary difference for a 5% level of significance was 2.2 the only significant variation occurred in the four minute period compared with the one and two minute levels.

Starch Content of Gluten

The variation in starch content of the gluten paralleled closely that of starch recovery owing to the close inverse correlation between the two. Since the four minute agitation time gave further reduction in starch content over the three minute period it would be desirable to use the longer time in spite of the fact that the three minute period was sufficient for optimum starch recovery.

As in starch recovery, a significant interaction existed between agitation time and volume of mixing water. As agitation time increased, starch removal from the gluten was accelerated by using a larger volume of mixing water.

Purification of the Crude Gluten

The dried gluten from the separation contained 20 to 30% starch. Since a gluten with less starch would be desirable for most purposes, except perhaps direct food uses, a method was devised for further removal of the starch.

Shredding and rewashing the wet crude gluten substantially reduced its starch content. The first pass gave proportionately greater reduction than the second. The finer sieve plate gave a larger washing surface and brought about greater reductions in starch although the processing capacity was reduced by half. Results are given in Table VIII.

TABLE VIII
DATA ON THE REFINEMENT OF CRUDE GLUTEN BY MEANS
OF SHREDDING AND WASHING

Gluten sample	Shredding treatment	Diameter of holes in sieve plate, in.	Starch in gluten, %
1	None	5/32	23.8
	1st pass		14.2
	2nd pass		12.4
2	None	1/16	23.2
	1st pass		11.9
	2nd pass		9.9

This method of refining, in addition to reducing the starch content of the gluten, increased the starch recovery by 2 to 3%.

Recommended Procedure

On the basis of the results obtained in this study, the following procedure is recommended: Patent flour is mixed with 80 to 85% (the actual quantity depending on the moisture content and gluten characteristics of the flour) of its weight of water. The dough, at a temperature of 25° to 30° C., is mixed for 15 to 25 min. It is then agitated in twice its own weight of water at the same temperature by means of a reversing paddle stirrer for approximately three minutes or until the curds are about 1 cm. in diameter. The mixture is pumped to a gyrating screen of about 150 mesh in order to separate the crude gluten curds from the starch milk.

The starch content of the gluten is readily reduced by shredding the gluten into water and subjecting the mixture to the agitation and screening procedure used in the primary separation.

The procedure recommended above may be subjected to minor variation, since the separation may be accomplished by means of various types and modifications of equipment depending on the size of operation. There is no doubt, however, that the general conditions described above are applicable to large-scale processing.

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A SMALL SEPARATOR FOR THE RECOVERY OF MILKWEED FLOSS AND SEED

II. PERFORMANCE STANDARDIZATION¹

By D. H. HAMLY²

Abstract

Standardization work with a small floss separator is discussed. The standardization of the mode of separation and of the treatment of pods make possible the demonstration of the existence of marked differences in the products from several clone varieties of milkweed.

During the development of the small separator for milkweed floss and seed described in Part I (1), many experimental threshings were carried out. Though these were performed to determine the effectiveness of variations of the techniques used in threshing, it was noted that the clone variety, maturity, moisture content, and storage treatment of the pods influenced the results. It was concluded that many operating conditions would have to be controlled and the products weighed if the separator's performance was to be properly evaluated.

In order to determine these conditions, 10 controlled experiments have been carried out in which the fractions of floss, seed, and hull materials found in the several traps were assayed after the threshing of pods of several different kinds and states. Experiment 11 was carried out under similar conditions to obtain a large quantity of floss from random collected pods. The first five experiments determined the standard conditions of operation, which have been described previously. In the remaining experiments the kind of pod material was changed for each experiment. Table I summarizes the results of Experiments 5 to 10, in which the standardized conditions of operation were maintained, except that in No. 6 the rate of feeding and air flow were higher than standard, and in No. 7 the rate of feeding was lower than standard.

The Material

The milkweed pods were collected from a large stand of wild plants that have been established for more than six years in grass sod on an abandoned farm at Bayview Ridge, Toronto. They are found distributed in distinctly separated clones, which in many cases show marked differences in character of growth. Sixteen clones have been staked and are under observation for collection of data for further studies. Pods used in Experiment 1 were collected in early October, 1943. They were improperly dried and stored and had developed conspicuous mildew. The other pods were picked in late September and early October, 1944, dried in the open in the onion bags used

¹ Manuscript received June 16, 1945.

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TABLE I
PERCENTAGE SEPARATION OF FLOSS, SEED, AND HULL MATERIALS

Fraction	Experiment number and source of material					
	5 Random	6 Random	7 Clone 9	8 Clone 5	9 Clone 7	10 Clone 8
Floss—Total	25.1	26.0	21.2	25.2	21.9	20.5
First Operation	89.0	81.6	75.2	77.7	91.6	90.5
Separator	8.9	13.7	21.9	16.7	6.2	7.1
Beater Trap	0	0.2	0.6	0.4	0.4	0.9
Seed Trap	0	0	0	0.1	0.2	1.1
Hull Trap	2.1	4.5	2.3	5.1	1.6	0.4
Seed—Total	36.1	36.1	44.4	32.9	38.6	34.6
Floss Trap	0	0	0	0	0	0
Separator	0.9	2.1	0.8	1.5	0.3	0.7
Beater Trap	94.4	90.0	98.7	91.2	95.2	95.1
Seed Trap	3.6	4.9	0.5	3.6	2.7	2.5
Hull Trap	1.1	3.0	0.02	3.7	1.8	1.7
Hull material—Total	38.8	37.9	34.4	39.5	39.5	44.9
Floss Trap	0	0	0	0	0	0
Separator	5.4	14.6	9.9	12.5	8.6	3.8
Beater Trap	28.8	23.9	33.0	32.8	56.3	43.8
Seed Trap	3.9	2.4	3.7	5.7	2.5	3.9
Hull Trap	61.9	59.1	53.4	49.0	32.6	48.5

for collection, and then stored under cover at room temperature. Those used in Experiments 2 to 6 and 11 were random collected. The pods used in the other threshings were collected from staked clones. Pods from Clone 9 were used in Experiment 7, from Clone 5 in Experiment 8, from Clone 7 in Experiment 9, and from Clone 8 in Experiment 10.

The Method of Assay

The contents of each trap were assayed according to the following plan:

Floss Trap.—Seed content of floss bag determined by count as heads of floss carrying seeds entered floss bag.

Beater Trap.—Broken floss removed by hand and weighed. Remaining materials screened through $\frac{1}{4}$ in. and $\frac{1}{8}$ in. mesh screens into three fractions (hull materials, seed and debris, seed wings and fine debris) and weighed. From the middle fraction a sample was quartered out, cleaned in a fanning mill, reduced in size by passes through a Jones Sampler, and finally hand cleaned to seed. From weighings taken at the end of each operation a calculation was made of the floss, seed, and hull materials in the trap.

Seed Trap.—This material was assayed as described under Beater Trap.

Separating Tower Trap.—Material repeatedly re-threshed by being fed directly into the beater until entirely distributed to beater, seed, hull, and floss traps. Beater and seed trap fractions bulked and assayed as described under Beater Trap. Hull trap material was then entirely hull materials and was recorded as such.

Hull Trap.—This material was assayed in the same manner as the Separating Tower material.

Separating Efficiency

As is apparent from the distinctly different ratios of floss, seed, and hull materials in Experiments 7, 8, 9, and 10 (Table I) the percentage recovery of any one component does not give a true index of the separator's efficiency. On the other hand the same assay data can be used to show the efficiency, that is, the completeness of any separation, for the first operation. The following ratios are employed in Table II: floss to total floss, beater and seed trap seed to total seed, debris in seed to beater and seed trap materials, floss or seed in hull trap materials, and hull materials in hull trap to total materials in hull trap. The values are calculated on a weight percentage basis. The minimum, maximum, average, and maximum deviation from the average are shown to demonstrate the importance of the variation present.

The maximum deviations from the average percentage efficiency values for the separation of a given component vary from 2.8 to 17.5% and are a measure of the sensitivity of the separator to differences in the kind and conditions of the pods. The deviation is large in hull materials, moderate in floss, and small (for practical purposes zero) in seed from hull materials.

The large deviation in the case of hull materials is correlated with the biological fact that there are conspicuous differences in milkweed pod hulls that affect the character of the separation. For example, some hulls are thin and tough, and some are thick and friable.

The same correlation exists, though to a lesser degree, in the responses of the separator to differences in the character of the floss.

Breakage occurs in both floss and hull materials to a moderate degree and this breakage might be used as a measure of efficiency; this is not really practical since quantitative measurements are difficult. The use of breakage as a gauge is also not desirable since it varies with the moisture content of the pods. This has been shown by Experiment 11 and by a series of experiments in which clone variety floss from Experiments 7, 8, 9, and 10 has been tested for breakage while exposed to different levels of humidity. In Experiment 11* pods had been stored in an atmosphere of 50 to 60% relative humidity instead of the usual 25%. This resulted in such a conspicuous decrease in breakage of both floss and hull materials that it is intended to use this humidity level as standard in future work when breakage needs to be at the minimum level.

Other factors affecting conditions are mildew and immaturity. These affect the degree of separation, and hence efficiency, in a way similar to variations in water content. They are, however, abnormal and are not factors in pods picked at the proper stage and dried and stored as recommended.

* A complete assay was not made of this large threshing. The estimated proportions of the separated fractions are floss—24%, seed—34%, hull materials—42%. Quality is high as indicated by low proportions of seed, debris, fragmented floss heads, and broken floss fibres.

TABLE II
PERCENTAGE EFFICIENCY OF SEPARATION

Material	Weight ratio	Experiment number and source of material							Summary		
		5 Random	6 Random	7 Clone 9	8 Clone 5	9 Clone 7	10 Clone 8	Min.	Max.	Av.	Max. dev.
Floss	$\frac{\text{Trapped floss}}{\text{Total floss}}$	89.0	81.5	75.3	77.8	91.5	90.5	75.3	91.5	83.3	+ 8.2
	$\frac{\text{Beater and seed trap seed}}{\text{Total seed}}$	98.0	95.0	99.2	94.7	98.0	97.5	94.7	98.0	97.1	- 2.4
Seed	$\frac{\text{Debris}}{\text{Beater and seed trap mat.}}$	26.4	21.5	28.7	34.1	41.4	39.3	21.5	41.4	31.9	-10.4
	$\frac{\text{H.M. in hull trap}}{\text{Total hull material}}$	61.8	55.2	53.3	49.0	32.6	48.5	32.6	61.8	50.1	-17.5
Floss in H.M.	$\frac{\text{Floss}}{\text{Hull trap materials}}$	2.3	5.6	2.6	6.2	2.6	0.4	0.4	6.2	3.2	+ 2.8
	$\frac{\text{Seed}}{\text{Hull trap materials}}$	1.7	5.2	0.4	6.0	5.3	2.8	0.4	6.0	3.5	- 3.1
H.M. in hull trap	$100\% - (\%F + \%S)$	96.0	89.2	97.0	87.8	92.1	96.8	87.8	97.0	93.3	- 5.5

NOTE:—($\%F + \%S$) is the sum of the percentages of floss and seed trapped with the Hull Materials, $100\% - (\%F + \%S)$ is a difference equivalent to the Hull Materials trapped with the floss and seed in the Hull Trap.

Conclusion

The development and standardization work with the milkweed floss separator has indicated that three sets of factors are involved in threshing milkweed pods. Two of these, the mode of operation and the state of the pods, have been standardized in order that a high yield of good quality products can be obtained with regularity. The standardization of these factors has made possible the demonstration of the third factor, i.e., marked differences in the products from clone variety milkweed pods.

The laboratory separator (Model IV) for the recovery of milkweed floss and seed has given efficient performance. Significant quantitative differences in the floss and seed of clone variety milkweeds have been demonstrated. In the continuous operation separator (Model V) the same high efficiency is to be retained in a machine of 50% greater capacity. It was designed to permit transport by small truck or trailer, an essential factor in the economical reclamation of wild milkweed growing in marginal farm areas far from the places of utilization.

Reference

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THE THERMAL PROPERTIES OF WHEAT IN BULK¹

By J. D. BABBITT²

Abstract

By enclosing wheat in a cylinder and heating by means of a wire stretched along the axis, the thermal conductivity k and the thermal diffusivity κ are determined. From the values obtained for these two quantities the specific heat is calculated.

The following values are obtained: $k = 0.00036$ cal. per sq. cm. per sec. per $\frac{^{\circ}\text{C.}}{\text{cm.}}$; $\kappa = 0.00115$ cm.² per sec., and $c = 0.37$ cal. per gm. per $^{\circ}\text{C.}$ By the use of these values of the thermal constants, an estimate is made of the effect of external changes on the temperature within stored wheat.

A few years ago during the course of an investigation into the storage of wheat, it became apparent to the author that complex temperature changes took place within stored wheat during the course of the year and that under certain conditions very large temperature gradients were established. At that time some attempt was made to calculate the temperature change that might be expected to take place within the wheat, but it proved difficult to obtain a satisfactory agreement between the calculated and observed temperatures. In that work the value used for the thermal diffusivity was obtained from estimated values of the thermal conductivity and the specific heat. It was probable that the discrepancy between observed and calculated temperatures arose from the failure of the theoretical equation to fit the actual conditions. There was, however, some doubt about the accuracy of the value used for the diffusivity, and it was decided that it would be worth while to measure this quantity directly since the measurement could be made quite simply and the experiment would, at the same time, furnish additional information on the thermal properties of wheat.

With this idea in mind the apparatus shown in Fig. 1 was designed. The wheat is contained in a galvanized iron cylinder, 1 ft. in diameter and 2 ft. in height. A heating element of No. 26 gauge chromel C wire is stretched along the axis of the cylinder and three sets of thermocouples are spaced across parallel diameters at different heights. Since the temperature depends only on the radius the temperature distribution within the wheat at any time can be completely determined by means of these thermocouples. An experiment is started with the wheat at a uniform temperature equal to that of the surroundings. A current is then sent through the heating element and the temperatures at the different thermocouples are measured at intervals until thermal equilibrium is attained. The final distribution of temperature is a function of the thermal conductivity and gives a measure of this quantity; the rate of change of the temperature distribution allows us to determine the

¹ Manuscript received in original form May 10, 1945, and as revised, July 26, 1945.

Contribution from the Division of Physics and Electrical Engineering, National Research Laboratories, Ottawa, Canada. Issued as N.R.C. No. 1338.

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thermal diffusivity; from a knowledge of these two quantities the specific heat may be calculated. In this way all the thermal properties of the wheat can be determined.

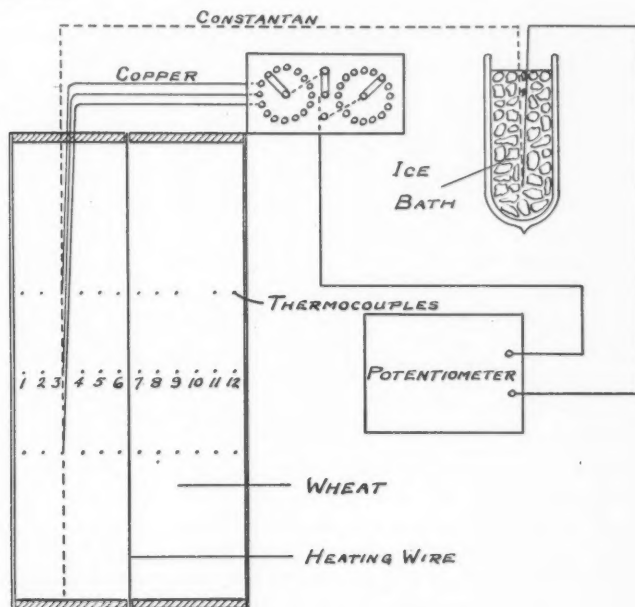


FIG 1. Diagram of apparatus.

Theoretical Considerations

According to Carslaw (1), the temperature v at any point in a right circular cylinder in which the initial temperature distribution is a function of the radius only ($v = f(r)$) and whose surface is maintained at zero temperature is given by

$$v = \sum_1^{\infty} A_n J_0(\alpha_n r) e^{-\alpha_n^2 \kappa t}. \quad (1)$$

Here $J_0(\alpha_n r)$ is Bessel's function of order zero of the first kind and the α 's are roots of the equation $J_0(\alpha_n a) = 0$. A_n is a constant that is determined by the assumption that the initial temperature distribution $f(r)$ can be expanded into the series

$$f(r) = A_1 J_0(\alpha_1 r) + A_2 J_0(\alpha_2 r) + \dots, \quad (2)$$

κ is the thermal diffusivity, and t the time.

In the experiments described in this paper the conditions are the reverse of those assumed above; the experiment starts with the wheat at a constant and uniform temperature equal to that of the surroundings and ends with a

temperature distribution independent of the time and dependent only on the radius. To satisfy these conditions, Equation (1) should be modified to

$$v = \sum_1^{\infty} A_n J_0(\alpha_n r) (1 - e^{-\alpha_n^2 t}). \quad (3)$$

The values of A_n can be determined from the boundary conditions by multiplying both sides of Equation (2) by $rJ_0(\alpha_n r)dr$ and integrating from 0 to a . Then, since

$$\int_0^a r J_0(\alpha_m r) J_0(\alpha_n r) dr = 0$$

and

$$\int_0^a r [J_0(\alpha_n r)]^2 dr = \frac{a^2}{2} [J'_0(\alpha_n a)]^2,$$

where

$$J'_0(\alpha a) = \left[\frac{d}{dr} J_0(r) \right]_{r=\alpha a}, \quad (4)$$

we obtain for A_n the expression

$$A_n = \frac{\int_0^a r f(r) J_0(\alpha_n r) dr}{\frac{a^2}{2} [J'_0(\alpha a)]^2}. \quad (5)$$

For a cylinder heated by a wire coincident with the axis, the final equilibrium temperature distribution may be represented by an equation of the form

$$v = f(r) = B + C \log_e r, \quad (6)$$

where B and C are constants.

When we substitute this value for $f(r)$ in the numerator of Equation (5) and evaluate the denominator by Equation (4) we obtain for A_n the expression

$$A_n = \frac{B \frac{a}{\alpha_n} J_1(\alpha_n a) + C \left[\frac{a}{\alpha_n} J_1(\alpha_n a) \log_e a - \frac{1}{\alpha_n^2} \right]}{\frac{a^2}{2} J_1(\alpha_n a)^2}. \quad (7)$$

We have, therefore, the following equation for the temperature at any point within the cylinder at a time t measured from the instant of turning on the heating current:

$$v = \sum_1^{\infty} \frac{B \frac{a}{\alpha_n} J_1(\alpha_n a) + C \left[\frac{a}{\alpha_n} J_1(\alpha_n a) \log_e a - \frac{1}{\alpha_n^2} \right]}{\frac{a^2}{2} J_1(\alpha_n a)^2} (1 - e^{-\alpha_n^2 t}) J_0(\alpha_n r). \quad (8)$$

In this equation, B and C can be determined from the equilibrium curve and the α_n 's are given by the known solutions of the equation $J_0(\alpha_n a) = 0$. The equation thus gives us a means of determining the diffusivity κ .

Experimental Results

The wheat used in these tests was a No. 1 seed Marquis wheat, and the moisture content at the beginning of the test was 9.2% of the dry weight. In Table I are given the particulars of a typical run. The body of the table gives the temperatures at the different positions across the sample for the time intervals shown in the first column. The numbers 1 to 12 at the head of the columns indicate the positions of the thermocouples, which were numbered from left to right across the cylinder. Couple No. 1 was one-half inch from the surface and the other couples were spaced at intervals of one inch. The heating wire was halfway between couples No. 6 and 7. The readings for only the central row of thermocouples are recorded as no significant difference was found in the temperature at the three positions. The room temperature during the experiment was 79.2° F. and all temperatures in the table have been converted to degrees above room temperature. This is in agreement with Equation (8), where the temperature of the environment is assumed to be zero.

It is to be noted that the temperatures in the table are not accurately symmetrical about the midpoint and that small fluctuations occur, especially in those thermocouples nearest the surface. There are three reasons for this.

(i) Variations in the temperature of the room in which the experiment was carried out. The room was held as closely as possible at a steady temperature by means of electrical heaters controlled by a thermostat, but uncontrollable changes occurred in the radiation from the windows and walls. The half of the cylinder corresponding to the right-hand side of Table I faced a blank wall and was in this way protected somewhat from these variations. At one point in the experiment the room temperature dropped several degrees owing to failure of the thermostat, and the effect of this can be seen in the readings at 47 hr. 10 min. and those immediately following.

(ii) Variations in the heating current in the wire, and

(iii) Inaccuracies in the placing of the thermocouples.

The errors introduced by (iii) would be most apparent near the centre where the temperature gradient is greatest.

The experimental data from Table I have been plotted in Fig. 2, which shows the progressive movement of the heat through the cylinder. These data are represented in a different manner in Fig. 3, in which the change of temperature with time at any one point is plotted. The measurements from the right-hand side of Table I (i.e., thermocouples Nos. 7 to 12) are represented by the circles. These thermocouples have been chosen as giving more consistent and reliable results than Nos. 1 to 6. The crosses in Fig. 3 represent measurements from another experiment, which was performed under similar conditions.

It is interesting to note the final equilibrium temperature distribution. As mentioned above, this should be represented by an equation of the form $v = B + C \log r$. In Fig. 4 the various readings obtained for the final tem-

TABLE I
TEMPERATURE DISTRIBUTION AT DIFFERENT POINTS ACROSS THE CYLINDER AT VARIOUS TIME INTERVALS

$I = 1.01$ amp.: $V = 5.71$ v.: length of heating wire = 24.75 in.

Time		Thermocouple No.											
Hr.	Min.	1	2	3	4	5	6	7	8	9	10	11	12
Temperature, °F. above room temperature													
0	00	0.5	0.3	0.3	0.3	0.3	0.3	0.3	0.3	0.3	0.3	0.2	0.0
0	40	0.7	0.3	0.3	0.3	1.4	13.4	12.9	1.4	0.3	0.2	0.1	0.0
2	25	0.7	0.6	0.7	1.9	6.8	24.2	23.5	6.4	1.9	0.4	0.2	0.0
3	40	0.7	0.8	1.5	3.6	9.4	27.9	27.7	9.2	3.4	1.1	0.3	0.1
5	40	0.8	1.4	2.8	5.6	12.6	31.4	30.9	12.3	5.4	2.3	0.8	0.3
22	20	3.1	5.6	9.2	14.3	22.7	42.8	42.2	22.8	14.4	8.9	5.6	2.9
29	55	3.7	6.8	10.6	15.9	24.9	44.8	44.0	24.4	15.8	10.2	6.4	3.5
47	10	1.5	5.9	10.8	16.9	26.4	46.7	46.0	25.9	17.0	10.8	5.8	1.1*
48	50	3.1	6.2	10.6	16.8	26.3	46.8	46.0	25.9	16.8	10.2	5.7	2.5
51	10	4.2	6.9	10.9	16.8	26.3	46.5	45.8	25.8	16.7	10.5	6.4	3.3
70	45	4.3	7.6	11.9	17.8	27.2	47.9	47.3	26.8	17.8	11.8	7.4	4.1
95	05	4.5	7.8	12.3	18.3	28.1	49.4	48.7	27.6	18.3	12.0	7.6	4.1
116	20	4.3	7.9	12.4	18.6	28.8	50.7	49.4	27.8	18.2	11.8	7.4	3.9
119	10	4.3	7.9	12.4	18.6	28.3	48.6	48.2	27.8	18.5	12.2	7.6	4.1

* At this point the room temperature dropped several degrees owing to the failure of the thermostat.

peratures have been plotted against $\log_{10} r$, and a straight line has been drawn through the mean positions. It is apparent that, with the exception of the point corresponding to $r = 0.5$ in., the experimental readings can be accurately represented by a linear equation. The equation of the straight line in Fig. 4 is

$$V_r = 34.3 - 40.36 \log_{10} r, \quad (9)$$

where V_r is written to represent the equilibrium temperature at a point distance r from the axis. If natural logarithms are used, Equation (9) becomes

$$V_r = 34.3 - 17.53 \log_e r. \quad (10)$$

Thus $B = 34.3$ and $C = -17.53$.

It is to be noticed in Fig. 4 that the temperature does not become equal to zero at the surface of the cylinder ($r = 6.0$) but at the point $\log_{10} r = 0.8500$ ($r = 7.08$ in.). This is, of course, natural since a temperature drop must exist between the surface and the surrounding air. For a rigorous treatment we should not use the equation for the temperature distribution in a cylinder whose surface is maintained at temperature zero but that representing a cylinder whose surface is radiating into a medium at temperature zero. In order, however, to simplify the work we shall use Equation (1) and shall take as the surface of the cylinder not the true surface but an imaginary surface at a distance $r = a$, where $v = 0$. In this experiment, therefore, $a = 7.08$ in.

Equation (10) for the equilibrium temperature distribution can be used to determine the thermal conductivity of the wheat. According to Carslaw (cf. Reference (1, p. 114)), the equation governing the transmission of heat in the steady state for conditions as given above is

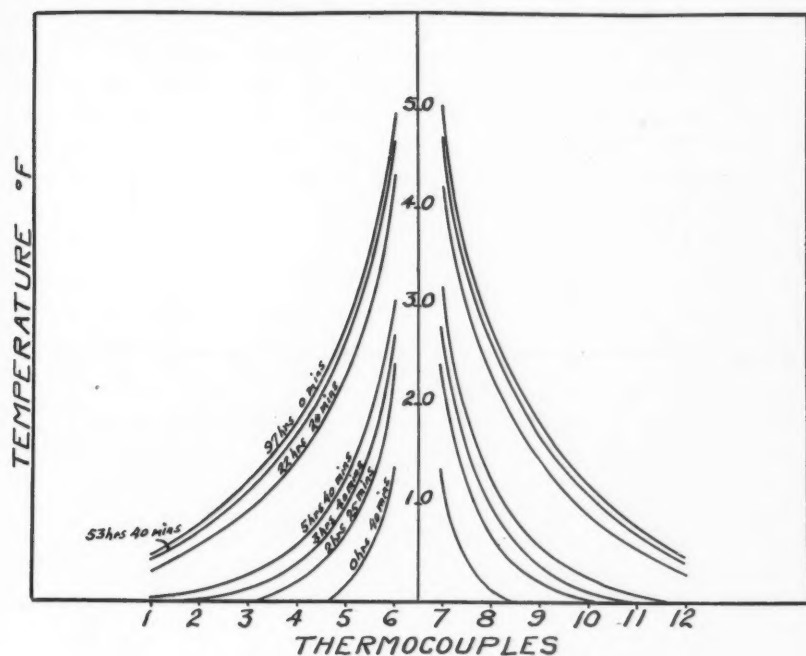


FIG. 2. Temperature distribution in cylinder.

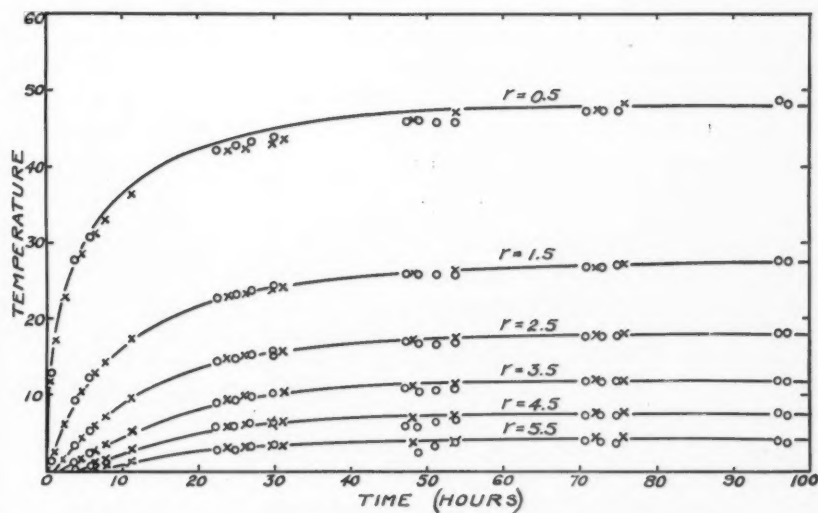


FIG. 3. Variation of temperature with time at different thermocouple stations.

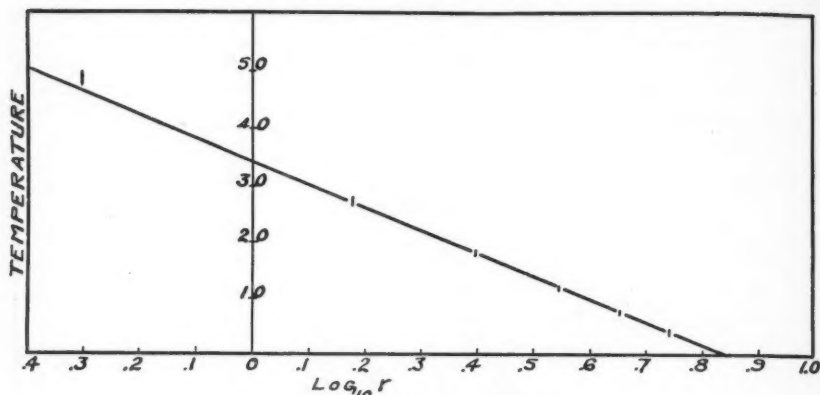


FIG. 4. Final temperature distribution.

$$V_r = \frac{I^2 R}{2\pi k} \log_e a - \frac{I^2 R}{2\pi k} \log_e r, \quad (11)$$

where I is the current and R the resistance per unit length of the heating element; k is the thermal conductivity. Comparing this equation with Equation (10) we see that

$$B = \frac{I^2 R}{2\pi k} \log_e a = 34.3 \quad \text{and} \quad C = \frac{I^2 R}{2\pi k} = 17.53,$$

and k can be obtained from either of these relations. The value obtained is 0.00036 cal. per sq. cm. per sec. per $\frac{^\circ\text{C}}{\text{cm}}$. The thermal conductivity of No. 1 Manitoba wheat (moisture content, 11.7%) has been given by T. A. Oxley (3) as 0.00036 , which is in complete agreement with the above.

Proceeding now with the equation for the temperature,

$$v = \sum_{n=1}^{\infty} A_n J_0(\alpha_n r) (1 - e^{-\alpha_n^2 kt}), \quad (3)$$

we evaluate A_n for the first five terms in the series. The following are obtained:

$$A_1 = 22.439; A_2 = 10.558; A_3 = 6.345; A_4 = 4.673; \text{ and } A_5 = 3.687.$$

Writing Equation (3) in full, we have

$$\begin{aligned} v = & 22.439(1 - e^{-\alpha_1^2 kt})J_0(\alpha_1 r) + 10.558(1 - e^{-\alpha_2^2 kt})J_0(\alpha_2 r) \\ & + 6.345(1 - e^{-\alpha_3^2 kt})J_0(\alpha_3 r) + 4.673(1 - e^{-\alpha_4^2 kt})J_0(\alpha_4 r) \\ & + 3.687(1 - e^{-\alpha_5^2 kt})J_0(\alpha_5 r) + \dots \quad (12) \end{aligned}$$

$$\begin{aligned} = & [22.439J_0(\alpha_1 r) + 10.558J_0(\alpha_2 r) + 6.345J_0(\alpha_3 r) \\ & + 4.673J_0(\alpha_4 r) + 3.687J_0(\alpha_5 r) + \dots] - [22.439e^{-\alpha_1^2 kt} \\ & J_0(\alpha_1 r) + 10.558e^{-\alpha_2^2 kt}J_0(\alpha_2 r) + 6.345e^{-\alpha_3^2 kt}J_0(\alpha_3 r) + \\ & 4.673e^{-\alpha_4^2 kt}J_0(\alpha_4 r) + 3.687e^{-\alpha_5^2 kt}J_0(\alpha_5 r) + \dots]. \quad (13) \end{aligned}$$

The expression in the first brackets on the right of Equation (13) is simply the expression for the equilibrium temperature obtained by making $t = \infty$. Since $J_0(\alpha_n r)$ is a periodic function, five terms are not sufficient to enable accurate evaluation of this expression. Instead we can use the experimental data given in Fig. 4. From this we obtain the following values for V_r :

r , in.	0.5	1.5	2.5	3.5	4.5	5.5	7.08
V_r , °F.	48.0	26.9	17.9	11.9	7.6	4.2	0.0

The equation for v can now be written

$$v = V_r - [22.439e^{-\alpha_1^2 t} J_0(\alpha_1 r) + 10.558e^{-\alpha_2^2 t} J_0(\alpha_2 r) + 6.345e^{-\alpha_3^2 t} J_0(\alpha_3 r) + 4.673e^{-\alpha_4^2 t} J_0(\alpha_4 r) + 3.687e^{-\alpha_5^2 t} J_0(\alpha_5 r) + \dots] \quad (14)$$

In practice only one or two terms of this expression are significant. To evaluate κ a point is taken on one of the curves in Fig. 3 where t is sufficiently large so that only the first term in the bracket need be considered. With the value of κ obtained in this way, Equation (14) can be used to evaluate v for any time t and any radius r .

The value of κ obtained is 0.00018 in.² per sec. or 0.00115 cm.² per sec. Using this value the curves drawn in Fig. 3 have been obtained and it is immediately evident that Equation (14) using $\kappa = 0.00018$ represents very closely the experimental results for all the curves except that for $r = 0.5$ in. It has already been mentioned that the point for $r = 0.5$ did not agree with the linear equation for the equilibrium temperature. The reason for the discrepancy is quite apparent; the heating wire was enclosed in pipe-clay tubing $\frac{1}{8}$ in. in diameter, and, since the theory assumes a heating element of negligible dimensions coincident with the axis, a noticeable deviation from theory at values of r as small as 0.5 is to be expected.

The value of the thermal diffusivity can be used in conjunction with the thermal conductivity to determine the specific heat of the wheat. We have the relation

$$c = \frac{k}{\kappa \rho} \quad (15)$$

where c is the specific heat and ρ the density.

The density of the wheat used in this experiment was measured and found to be 0.85 gm. per cc. (This is, of course, the bulk density and not the "true" density). Using this value and the values found above for k and κ we find the specific heat c to be equal to 0.37 cal. per gm. per °C.

The moisture content is 9.2%. In order to estimate the specific heat of dry wheat it is necessary to make an assumption about the contribution made by the adsorbed water. It is natural to assume that this water will have the same specific heat as water in the free state; there is, however, some doubt about this assumption. It is generally considered, and various experiments

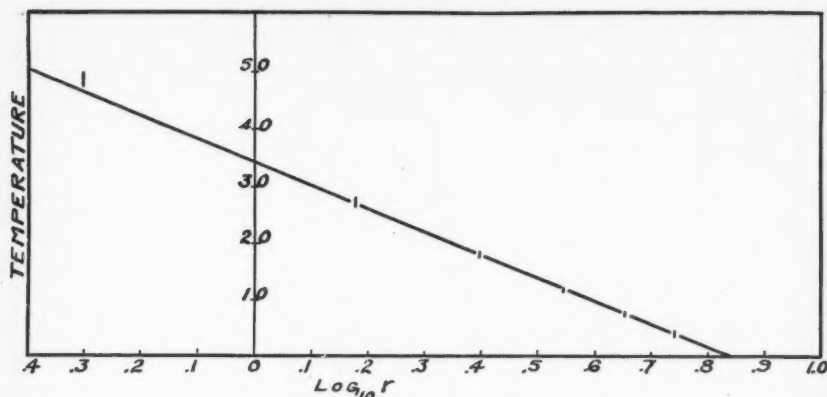


FIG. 4. Final temperature distribution.

$$V_r = \frac{I^2 R}{2\pi k} \log_e a - \frac{I^2 R}{2\pi k} \log_e r, \quad (11)$$

where I is the current and R the resistance per unit length of the heating element; k is the thermal conductivity. Comparing this equation with Equation (10) we see that

$$B = \frac{I^2 R}{2\pi k} \log_e a = 34.3, \quad \text{and} \quad C = \frac{I^2 R}{2\pi k} = 17.53,$$

and k can be obtained from either of these relations. The value obtained is 0.00036 cal. per sq. cm. per sec. per $\frac{^\circ\text{C.}}{\text{cm.}}$. The thermal conductivity of No. 1 Manitoba wheat (moisture content, 11.7%) has been given by T. A. Oxley (3) as 0.00036 , which is in complete agreement with the above.

Proceeding now with the equation for the temperature,

$$v = \sum_{n=1}^{\infty} A_n J_0(\alpha_n r) (1 - e^{-\alpha_n^2 \kappa t}), \quad (3)$$

we evaluate A_n for the first five terms in the series. The following are obtained:

$$A_1 = 22.439; A_2 = 10.558; A_3 = 6.345; A_4 = 4.673; \text{ and } A_5 = 3.687.$$

Writing Equation (3) in full, we have

$$\begin{aligned} v = & 22.439(1 - e^{-\alpha_1^2 \kappa t}) J_0(\alpha_1 r) + 10.558(1 - e^{-\alpha_2^2 \kappa t}) J_0(\alpha_2 r) \\ & + 6.345(1 - e^{-\alpha_3^2 \kappa t}) J_0(\alpha_3 r) + 4.673(1 - e^{-\alpha_4^2 \kappa t}) J_0(\alpha_4 r) \\ & + 3.687(1 - e^{-\alpha_5^2 \kappa t}) J_0(\alpha_5 r) + \dots \quad (12) \end{aligned}$$

$$\begin{aligned} = & [22.439 J_0(\alpha_1 r) + 10.558 J_0(\alpha_2 r) + 6.345 J_0(\alpha_3 r) \\ & + 4.673 J_0(\alpha_4 r) + 3.687 J_0(\alpha_5 r) + \dots] - [22.439 e^{-\alpha_1^2 \kappa t} \\ & J_0(\alpha_1 r) + 10.558 e^{-\alpha_2^2 \kappa t} J_0(\alpha_2 r) + 6.345 e^{-\alpha_3^2 \kappa t} J_0(\alpha_3 r) + \\ & 4.673 e^{-\alpha_4^2 \kappa t} J_0(\alpha_4 r) + 3.687 e^{-\alpha_5^2 \kappa t} J_0(\alpha_5 r) + \dots]. \quad (13) \end{aligned}$$

The expression in the first brackets on the right of Equation (13) is simply the expression for the equilibrium temperature obtained by making $t = \infty$. Since $J_0(\alpha_n r)$ is a periodic function, five terms are not sufficient to enable accurate evaluation of this expression. Instead we can use the experimental data given in Fig. 4. From this we obtain the following values for V_r :

r , in.	0.5	1.5	2.5	3.5	4.5	5.5	7.08
V_r , °F.	48.0	26.9	17.9	11.9	7.6	4.2	0.0

The equation for v can now be written

$$v = V_r - [22.439e^{-\alpha_1^2 \kappa t} J_0(\alpha_1 r) + 10.558e^{-\alpha_2^2 \kappa t} J_0(\alpha_2 r) + 6.345e^{-\alpha_3^2 \kappa t} J_0(\alpha_3 r) + 4.673e^{-\alpha_4^2 \kappa t} J_0(\alpha_4 r) + 3.687e^{-\alpha_5^2 \kappa t} J_0(\alpha_5 r) + \dots] \quad (14)$$

In practice only one or two terms of this expression are significant. To evaluate κ a point is taken on one of the curves in Fig. 3 where t is sufficiently large so that only the first term in the bracket need be considered. With the value of κ obtained in this way, Equation (14) can be used to evaluate v for any time t and any radius r .

The value of κ obtained is 0.00018 in.² per sec. or 0.00115 cm.² per sec. Using this value the curves drawn in Fig. 3 have been obtained and it is immediately evident that Equation (14) using $\kappa = 0.00018$ represents very closely the experimental results for all the curves except that for $r = 0.5$ in. It has already been mentioned that the point for $r = 0.5$ did not agree with the linear equation for the equilibrium temperature. The reason for the discrepancy is quite apparent; the heating wire was enclosed in pipe-clay tubing $\frac{1}{8}$ in. in diameter, and, since the theory assumes a heating element of negligible dimensions coincident with the axis, a noticeable deviation from theory at values of r as small as 0.5 is to be expected.

The value of the thermal diffusivity can be used in conjunction with the thermal conductivity to determine the specific heat of the wheat. We have the relation

$$c = \frac{k}{\kappa \rho} \quad (15)$$

where c is the specific heat and ρ the density.

The density of the wheat used in this experiment was measured and found to be 0.85 gm. per cc. (This is, of course, the bulk density and not the "true" density). Using this value and the values found above for k and κ we find the specific heat c to be equal to 0.37 cal. per gm. per °C.

The moisture content is 9.2%. In order to estimate the specific heat of dry wheat it is necessary to make an assumption about the contribution made by the adsorbed water. It is natural to assume that this water will have the same specific heat as water in the free state; there is, however, some doubt about this assumption. It is generally considered, and various experiments

have indicated, that water molecules are bound more strongly by adsorbing materials than by a free water surface and have not, therefore, the freedom of motion possessed by molecules in a liquid. In other words, the physical state of adsorbed water may be considered as partaking of some of the properties of a solid and it might be more appropriate to use the specific heat of ice rather than that of water. On the other hand, Shipley, Campbell, and Maass (4) have measured the specific heat of water adsorbed on cellulose and found no indications that, in this respect, the water behaved as a solid. At this point, therefore, notwithstanding the theoretical considerations advanced above, we shall consider the adsorbed water as possessing the specific heat of liquid water and calculate c for dry wheat on this basis. The figure obtained is 0.31 cal. per gm. per °C.

The literature on specific heats does not give any data for wheat but Winkler and Geddes (5) have determined c for wheat flour and obtained a value of 0.397 cal. per gm. per °C. Their determination was made by a "method of mixtures" in which water at a higher temperature was added to a dough of flour and water. They assumed what is equivalent to our assumption above—that all the water in the dough possessed the specific heat of liquid water. Their value of 0.397 is considerably larger than our value of 0.31 and all of the difference can hardly be attributed to the relatively small portion (approximately 13%) of bran that is eliminated from the wheat in the manufacture of flour. When, however, the assumptions made about the nature of the adsorbed water and the difference in the materials used and the methods employed are considered, a closer agreement could scarcely be expected.

As already stated the readings of the three sets of thermocouples agreed within the variations in the readings of the individual couples caused by inhomogeneities, uncertainty in position, and external temperature fluctuations. This agreement was a fairly good indication that convection currents did not play a predominant, or even a very large, part in the heat transfer. In order, however, to determine the effect of convection more accurately, the experiment was repeated with the axis of the cylinder horizontal and the row of thermocouples in a vertical direction. When the conditions within the wheat had come to equilibrium and the temperature distribution had been determined, the cylinder was turned on its axis through 180° so that the thermocouples that had formerly been below the axis were now above it. The thermal conditions were again allowed to come to equilibrium and the temperature distribution was determined once more. The average of the temperatures in the upper half of the cylinder in the two cases was now compared with the average found from the couples in the lower half. Any difference in the two sets can be attributed to the effect of convection since the reversal of the cylinder would eliminate all errors arising from individual couples. The results are given in Table II.

It is to be noted that there is approximately 0.5° F. temperature difference between the upper and lower half of the cylinder and that this difference may

TABLE II

DIFFERENCES IN TEMPERATURE OF THERMOCOUPLES ABOVE AND BELOW THE AXIS OF HORIZONTAL CYLINDER

$r =$	0.5	1.5	2.5	3.5	4.5	5.5
Mean top temp., °F.	46.0	26.6	17.6	11.9	7.9	4.5
Mean bottom temp., °F.	45.6	26.1	17.1	11.3	7.2	3.9
Difference, °F.	0.4	0.5	0.5	0.6	0.7	0.6

be assumed to be the effect of convection currents. It can therefore be stated that the proportion of the heat that is transferred by convection is a very small part of the total heat transferred. Oxley (3) from measurements on wheat in spherical containers with and without baffles has arrived at a similar conclusion.

Temperature Changes Occurring in Stored Wheat

When wheat is placed in storage in a grain elevator it is, since no effort is made to heat the elevator, exposed to any temperature changes taking place in the external atmosphere. The wheat when introduced into the storage space will, presumably, be at a uniform temperature that will not, in general, be the same as the mean annual temperature of the environment. The temperature of the wheat will follow changes in the external temperature and it will be interesting to use the values of the thermal properties determined above in order to study how the external temperature changes will affect the temperature of the wheat.

It will be assumed in what follows that there is no source of heat within the wheat itself. This is, of course, not true since heat will be generated by kernel respiration, mould growth, and insect infestation. In general under safe storage conditions, where the temperature is moderate and the moisture content below 14.5%, this internal source of heat is not sufficient to influence appreciably the temperature of the wheat, and for our purposes we may treat the grain as a mass of inanimate material. Under these conditions all temperature changes taking place in the wheat will originate in the external atmosphere and will enter the wheat through the surface.

The problem that arises when an attempt is made to estimate the temperature variation of a mass of wheat over a period of time is very similar to the problem of the temperature changes occurring in the crust of the earth. The temperature of the wheat, like that of the earth, will more or less closely follow the changes in the temperature of the external atmosphere and we shall expect to find, within the wheat, temperature changes corresponding to the daily variation of temperature from night to day and also much slower and greater variations due to the seasonal change from summer to winter. The mathematical theory of these periodic variations has been worked out for the surface of the earth and substantial agreement has been found with

measured temperatures. Besides the periodic changes there will be, in general, a slow change of temperature with time owing to the fact that the temperature at which the grain is introduced into the bin will not be equal to the mean external temperature. This effect corresponds to those secular changes that have given rise to the temperature gradient in the earth and that Lord Kelvin attributed to the cooling of the earth from its initial liquid state and on which he based his estimate of the age of the earth.

In order to apply the mathematical formulae governing these temperature changes we consider the mass of wheat as a semi-infinite body bounded at the surface by the plane $x = 0$ and extending to infinity in the direction of x positive. By this simplification we are concerned only with temperature changes in one direction since all planes parallel to $x = 0$ will have a uniform temperature. In practice the temperature changes are never confined to one dimension, but, in a centre bin of a grain elevator or a pile of wheat in a temporary storage where the lateral dimensions are large compared with the vertical, the temperature gradients in a horizontal direction are small and may be neglected.

As mentioned above, the three types of temperature change that will take place in our mass of wheat are:

- (1) The diurnal temperature variation corresponding to the difference in temperature between night and day,
- (2) The annual variation corresponding to the seasonal changes of temperature, and
- (3) The slow temperature change arising from the fact that the temperature of the wheat when placed in the bin does not correspond to the mean temperature of the surroundings and must therefore slowly approach that temperature.

We shall treat these three temperature changes in some detail in order to see how they will affect the temperature of stored wheat.

The Diurnal Temperature Wave

In order to study the effect of the diurnal temperature variation we assume that the changes in external temperature may be represented as a harmonic function of time. It is quite obvious that this is not true since the temperature changes occurring during the course of a day are very irregular and an extensive Fourier's series would be required to represent them adequately. By expressing the changes as a harmonic function we are using only the principal term of the Fourier's series where the period τ is equal to one day, but in this analysis we are interested only in general conclusions, and this simplification will be sufficient to indicate all the general features of the phenomenon. We assume also that this harmonic variation of temperature occurs at the surface of the wheat. In actual practice the temperature of the surface is governed by the fact that it is radiating into a medium whose temperature varies periodically, but here again our conclusions will not be affected. We

assume then that the surface temperature varies according to the equation

$$v_s = v_d \cos(\omega t - \epsilon), \quad (16)$$

where v_d is the amplitude of variation of temperature, ω the frequency which is related to the period τ by the equation $\omega = \frac{2\pi}{\tau}$ and ϵ is the phase angle.

The effect of this harmonic temperature variation at the surface is to superimpose on the temperature at the plane x a periodic temperature fluctuation given by

$$v = v_d e^{-\sqrt{\frac{\omega}{2K}} x} \cos\left(\omega t - \sqrt{\frac{\omega}{2K}} x - \epsilon\right). \quad (17)$$

Thus as x increases and we proceed away from the surface the amplitude of temperature change steadily decreases because of the exponential term and the phase of the wave changes because of the term $\sqrt{\frac{\omega}{2K}} x$. The wave-length λ of this temperature wave is equal to $2\sqrt{\pi K \tau}$ and the velocity with which it travels into the wheat is given by $2\sqrt{\frac{\pi K}{\tau}}$.

In order to ascertain the effect that this diurnal temperature wave has on the wheat we shall assume that the variation of the temperature at the surface between day and night is 20°F. ; v_d is then equal to 10° . With this assumption we find that 5.1 in. below the surface the amplitude of the variation has decreased to 1°F. Thus we can say that below five inches the effect of the daily temperature variation will be scarcely noticeable. Kelly (2) has already pointed out this fact from observations made at different depths within some wheat stored in a 1000 bushel round metal bin. It can be concluded, therefore, that the diurnal temperature variation would have very little effect on the storage of grain.

The Annual Temperature Variation

When we come to consider the annual temperature variation we find conditions very similar to the above except that the period is now 365 days and the amplitude of the temperature change is greater than that for the daily variation. We assume as before that the variation is harmonic and use for the equation of temperature change at the point x

$$v = v_a e^{-\sqrt{\frac{\omega}{2K}} x} \cos\left(\omega t - \sqrt{\frac{\omega}{2K}} x - \epsilon\right). \quad (18)$$

For Port Arthur the highest average temperature for a month is 73°F. (July) while the lowest is -4°F. (January and February). We assume then that a reasonable temperature range for wheat storage would be 77°F. and obtain a value of 38.5° for v_a . If we again calculate the depth at which the amplitude of variation has decreased to 1°F. we obtain this time an answer of 12 ft. 11 in. We can say, therefore, that the annual changes of temperature do not appreciably influence the temperature of stored wheat at a distance below the surface greater than 13 ft.

It is interesting to note the lag of the temperature wave at different depths within the wheat. This is given by the phase angle $\sqrt{\frac{\omega}{2k}} x$. When x is equal to 5.5 ft. the temperature wave is 90° out of phase. That is to say, that $5\frac{1}{2}$ ft. below the surface the temperature changes will occur three months after the corresponding change at the surface. The maximum in the temperature curve that takes place at the surface in July will not reach a point 5.5 ft. below the surface until October and it will be January before it gets to a depth of 11 ft.

The Secular Temperature Change

Finally we must consider the temperature change by which the temperature of the wheat slowly becomes equal to the mean temperature of the surroundings. To study this effect we have the equation given by Carslaw (1) for the temperature distribution in a semi-infinite solid that has an initial uniform temperature v_0 and whose surface $x = 0$ is maintained at the temperature zero. This expression is

$$v = \frac{2v_0}{\sqrt{\pi}} \int_0^{\frac{x}{2\sqrt{kt}}} e^{-\xi^2} d\xi. \quad (19)$$

If the surface temperature is v_s and not zero the equation becomes

$$v - v_s = \frac{2(v_0 - v_s)}{\sqrt{\pi}} \int_0^{\frac{x}{2\sqrt{kt}}} e^{-\xi^2} d\xi. \quad (20)$$

The definite integral in this equation is given in various tables and v can easily be evaluated.

In order to ascertain how this secular change will affect the conditions within stored wheat we shall take the hypothetical case of a bin filled initially with wheat at a temperature of 0° F. and exposed on its upper surface to a temperature of 36° F. This corresponds to the mean yearly temperature at Port Arthur.

The temperature distribution calculated from Equation (20) at intervals of three months up to the end of one year is shown in Fig. 5. It is interesting to note that at the end of three months there has been practically no change in the temperature below 10 ft. and even at the end of a year the temperature of the wheat below 20 ft. has increased less than 1° F.

It must be remembered that in practice the three thermal effects would be additive and no simple increase in temperature such as that shown in Fig. 5 would be obtained. The diurnal and annual temperature changes would have to be added to the curves in Fig. 5. The general conclusions, however, remain; the annual temperature variation is never greater than 1° F. below 13 ft. and the long term temperature change is negligible at this point for all periods less than six months; at the end of a year the temperature 20 ft. from the surface is practically unchanged and the wheat remains at the temperature at which it was put into the bin.

These points are important for the storage of wheat in large grain elevators or in large masses since it shows that the temperature changes arising outside the grain itself are largely confined to the surface layers and that at great depths the exterior temperature has no effect.

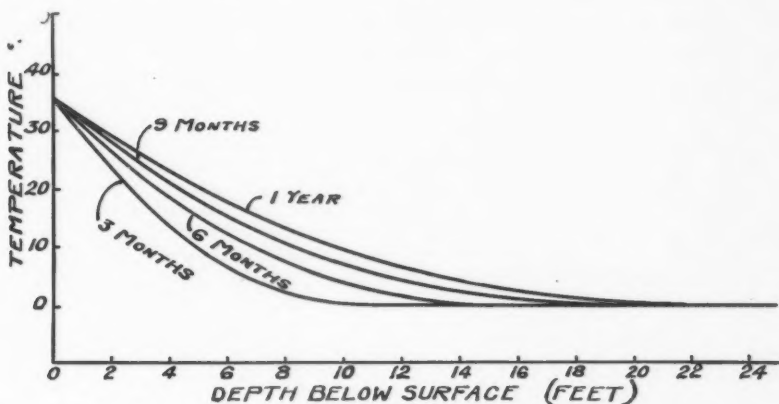


FIG. 5. Secular temperature changes.

Acknowledgment

The author wishes to express his appreciation of the assistance of Mr. C. St. Jacques, Laboratory Assistant at the National Research Laboratories, who has constructed the apparatus used in this experiment and assisted with many of the measurements.

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CHEMICAL ASPECTS OF THE APPLICATION OF DUST-LAYING OILS TO WOOL¹

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Abstract

The treatment of woollen blankets with dust-laying oils for the purpose of reducing dust-borne infection may be accomplished by the use of positively charged emulsions of oil in water. The chemical aspects of this process have been investigated and the influence of the various factors that affect the rate of exhaustion of the treating bath has been studied. It was found that the rate of exhaustion is increased by (a) raising the temperature, (b) raising the pH, and (c) reducing the concentration of emulsifying agent to the minimum amount necessary for stable emulsification of the oil. Traces of soap remaining in the wool after laundering cause a reversal of the electrical charge on the emulsion droplets and therefore prevent complete exhaustion of the treating bath. Suggestions are made regarding the practical application of the process.

The treatment of hospital bed-clothes with dust-laying oils has recently received considerable attention in the medical literature. Thomas and van den Ende (10) have shown that the impregnation of bed-clothes with 3 to 7% of liquid paraffin oil results in a 95% reduction in the number of organisms distributed into the air during bed-making. Two methods for applying the oil were used in the earlier trials: (a) impregnation with oil dissolved in a volatile organic solvent (11), and (b) impregnation with concentrated oil-in-water emulsions (12).

Harwood, Powney, and Edwards (7) have developed an improved process, in which use is made of a cation-active emulsifying agent that imparts a positive charge to the oil droplets of the emulsion. When negatively charged material (e.g., wool) is immersed in this emulsion, complete exhaustion of the oil occurs owing to electrostatic attraction. This process possesses several advantages over the former methods, viz.:

- (a) There is no waste of oil, since the emulsion is exhausted completely;
- (b) The degree of oiling can be controlled accurately;
- (c) No additional equipment is required since the oiling is carried out in the laundry washwheel at the conclusion of the normal laundering procedure. The residual liquor, being essentially clear water, is discarded.

The present paper deals with some of the chemical aspects of this process, viz., the effects of temperature and pH of the emulsion, of concentration of the emulsifying agent and of the oil, and of the presence of interfering substances (e.g., soaps) on the rate of exhaustion of the oil from the treating bath.

¹ Manuscript received June 23, 1945.

Contribution from the Department of Chemistry, National Research Laboratories, Ottawa, Canada. Issued as N.R.C. No. 1337.

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Experimental

Material Used

Undyed wool serge was used as the test material. It was thoroughly rinsed in warm distilled water, dried, and extracted in a Soxhlet apparatus with 95% ethyl alcohol for 48 hr. The soap content determined by the method of King (2, 3) was found to be less than 0.01% calculated as sodium oleate.

A technical white oil (Marcol HX, supplied by Imperial Oil Company) was used throughout this work. This oil conforms to the following physical specifications:

Specific gravity at 60° F.	840/850
Colour Saybolt	+ 30 up
Closed cup flash	310° F. min.
Pour test	20° F. max.
Saybolt viscosity at 100° F.	75/85
Copper test	Passes
Taste	Tasteless
Odour	Odourless
U.S.P. Acid test	Passes

In addition, van den Ende and Thomas (12) have found this oil to be completely inert with regard to carcinogenic activity. An oil of this type is believed to be satisfactory for use in the commercial application of the oiling process, and hence it was felt that the results obtained might be of greater practical value if this oil were used rather than the more highly refined and more expensive oils of the medicinal type.

The emulsifying agent used was a commercial preparation of cetyl dimethyl benzyl ammonium chloride containing approximately 25% of active ingredient. For the sake of convenience in the following discussion, the term "emulsifying agent" will be used to denote the commercial preparation, and the symbol "E" will be used in referring to the chemical compound cetyl dimethyl benzyl ammonium chloride.

Distilled water was used throughout.

Preparation of Emulsion

A stock emulsion of the oil was made up as follows:

Hot water (140° to 160° F.)	250 ml.
Emulsifying agent	20 gm.
Oil	200 gm.
Cold water	500 ml.

The emulsifying agent was dissolved in the hot water and the oil stirred in. The mixture was then diluted with the cold water and passed through a colloid mill. On standing, this emulsion tended to "cream" but there was no separation of clear oil, and on gentle mixing an emulsion of uniform appearance was again obtained. This emulsion was used throughout, except in those cases in which the influence of varying concentration of emulsifying agent was studied.

Method of Oiling

The apparatus used consisted of a wide-mouthed 500 ml. Erlenmeyer flask, equipped with an electrically driven stirrer, and supported in a constant temperature water-bath, the temperature of which was controlled to within $\pm 0.2^\circ \text{C}$. The required volume of stock emulsion, diluted with 200 ml. of water, was measured into the flask and brought to constant temperature. A 25 ml. aliquot was pipetted out for determination of the initial oil content, and then 10 gm. of the prepared wool, cut into pieces about 1 in. square, was added. At intervals of 5, 10, 20, and 40 min., 25 ml. samples of the emulsion were removed for analysis. The initial and final pH of the emulsion was measured using a Beckman pH meter. The oil content of the sample was determined by extraction with three successive portions of petroleum ether (boiling range 30° to 60°C .). Isopropyl alcohol was added to break the emulsion and give a clear separation of the layers. After separation, the ether extracts were combined, filtered, and the filter paper was washed with an additional 25 ml. portion of ether. The bulk of the ether was distilled off, the residue was transferred to a weighed evaporating dish, and the remainder of the ether removed by evaporation over a water-bath. The results are expressed in terms of percentage reduction in emulsion concentration, viz.,

$$\frac{W - W_T}{W} \times 100 = \% \text{ reduction in emulsion concentration after } T \text{ minutes,}$$

where W = weight of oil recovered from initial 25 ml. aliquot,

W_T = weight of oil recovered from aliquot taken after T minutes.

Influence of Temperature

In order to study the influence of temperature on the rate of exhaustion of the emulsion, a series of runs, as described above, were carried out at temperatures of 25° , 40° , 55° , and 70°C ., using 10 ml. of the stock emulsion. An attempt was made to maintain the pH at a constant value by the use of buffered solutions, but it was found that the presence of the buffer caused coagulation of the emulsion. The initial pH was 4.0 and the final pH 6.5 to 6.6. The results are given in Fig. 1.

The exhaustion of oil from the emulsion is brought about by two means. The first of these is mechanical filtering out of the oil drops by the fabric, and is governed by the concentration of oil, the size of the drops, and the rate of agitation. The second means of exhaustion is the electrostatic attraction of the wool for the charged oil drops and is governed principally by the concentration of oil and the cataphoretic mobility of the drops. Powney and Wood (8) have shown that for suspensions in water of a medicinal mineral oil (Nujol), the change in mobility with temperature depends only on the viscosity of the water according to the relationship $u_1\eta_1 = u_2\eta_2$, where u represents the mobility of the drop and η the viscosity of the water. Since the viscosity decreases with increasing temperature, it follows that the mobility of the drops and hence the rate of exhaustion of the emulsion increases with increasing temperature. It is difficult to apply any mathematical treatment to the data

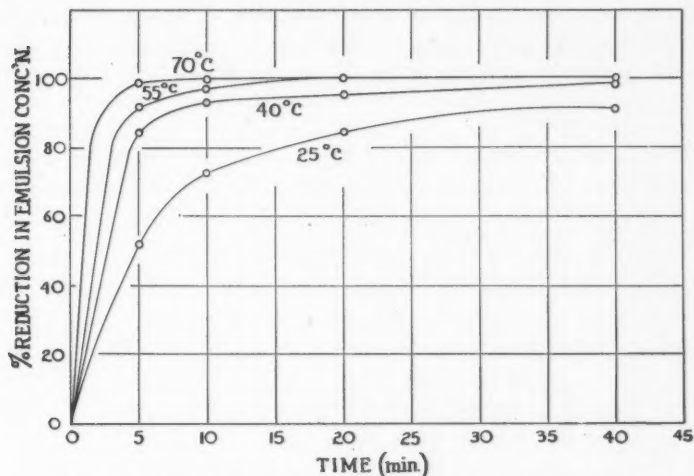


FIG. 1. Influence of temperature on the rate of emulsion exhaustion.

owing to the fact that the relative contributions of mechanical filtration and electrostatic attraction are unknown.

Influence of pH of the Emulsion

A series of runs similar to those previously described was carried out at a temperature of 40° C. using 10 ml. of the stock emulsion. The initial pH values of these emulsions were adjusted by the addition of dilute solutions of hydrochloric acid, sodium bicarbonate, or sodium carbonate. The results are given in Fig. 2.

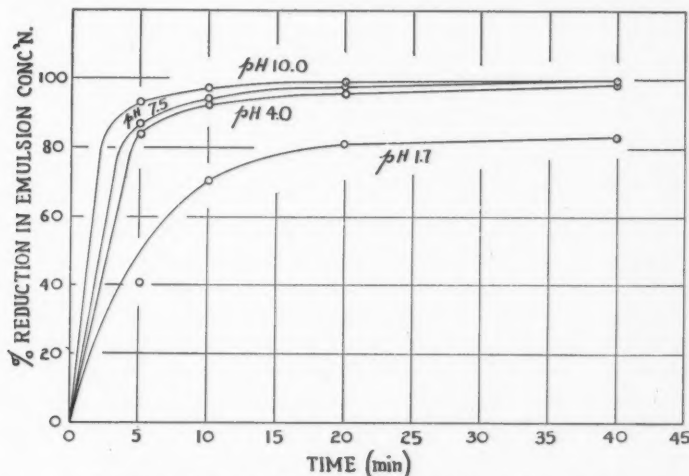


FIG. 2. Influence of pH on the rate of emulsion exhaustion.

The rate of exhaustion of oil from the emulsion due to electrostatic attraction depends on the magnitude and sign of the electrical charge on the wool. Harris (6) has shown that this charge is zero at pH 3.4 (the isoelectric point), that it increases positively as the pH is decreased below this point, and that it increases negatively as the pH is increased. It is, therefore, reasonable to assume that at pH values below 3.4, the pick-up of oil is due entirely to mechanical filtration, while at pH values above 3.4, the contribution of electrostatic attraction becomes increasingly greater.

Influence of Concentration of Emulsifying Agent

A series of stock emulsions were made up containing approximately 70% of oil and amounts of emulsifying agent equivalent to 2.5, 5, and 10% of the weight of the oil. Weights of these stock emulsions estimated to give initial concentrations comparable with those used previously were diluted with 200 ml. of water and experiments were carried out at 40° C. as described above. The results obtained are given in Fig. 3.

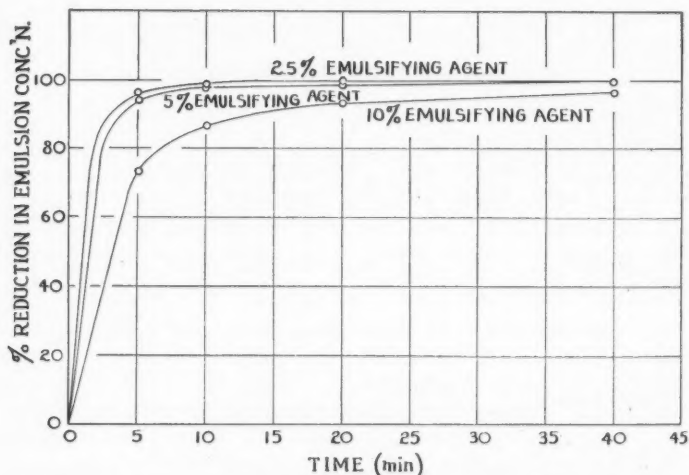


FIG. 3. Influence of concentration of emulsifying agent on the rate of emulsion exhaustion.

The stability of an emulsion is a function of the size of the dispersed droplets. Other things being equal, emulsions containing large drops will separate rapidly, while emulsions containing small drops will separate slowly. Schulman and Cockbain (9) state that drops of about 4μ diameter are sufficiently small to give an emulsion of good stability. Griffin (5), working with emulsions stabilized with soap, concludes that an emulsion is stable when a monomolecular film of soap is formed at the interface, but when there is not sufficient soap present to form a monomolecular layer, the emulsion is unstable. If excess soap is present, it remains dissolved in the aqueous phase and has little influence on the stability of the emulsion.

In view of the above discussion and provided the surface area occupied per molecule of the emulsifying agent is known, it is possible to calculate the minimum ratio of emulsifying agent to oil necessary to produce a stable emulsion. Unfortunately this information is not available for the emulsifying agent used in the present work, but data are given in the literature for a number of other emulsifying agents. Griffin (5) found 48 \AA^2 per molecule for sodium oleate, 27 for potassium stearate, and 30 for potassium palmitate. Fischer and Harkins (4), studying emulsions of paraffin oil, estimated that the area per molecule of soap in the interfacial film lies in general between 24 and 38 \AA^2 .

Considering 10 gm. of oil of specific gravity 0.84 and an average drop diameter of 4μ , the total surface area is $17.85 \times 10^{20} \text{ \AA}^2$. Assuming a probable value of 30 \AA^2 for the area occupied by one molecule of *E*, the weight of *E* required to saturate this surface area is 0.0386 gm. or 0.386% of the weight of the oil. Since the commercial product contains approximately 25% of *E*, the weight of emulsifying agent required is $4 \times 0.386 = 1.544\%$ of the weight of the oil. Hence, to produce a stable emulsion, the emulsifying agent must be present in an amount equal to at least 1.5% of the weight of the oil. It was found experimentally that emulsions containing 2.5% (based on the weight of oil) of the emulsifying agent were stable, whereas those containing 1.25% were not stable. This seems to justify the assumption of a molecular area of approximately 30 \AA^2 for *E*.

The theory that an amount of emulsifying agent in excess of this minimum requirement has little effect on the stability of the emulsion was confirmed by the fact that emulsions containing 2.5, 5, and 10% of emulsifying agent, based on the weight of oil, showed no differences in stability as judged visually. In the use of emulsions for the treatment of wool, however, too great an excess of emulsifying agent should be avoided since the free molecules dissolved in the aqueous phase are picked up rapidly by the wool, tending to saturate it, and thus limit its capacity for the adsorption of oil. This is shown in Fig. 3, where the emulsion containing 10% of emulsifying agent is seen to exhaust more slowly than those containing lesser amounts.

Influence of Concentration of Oil

The influence of the initial concentration of oil on the rate of exhaustion of emulsions at 40°C . was determined in the manner described previously, using 5, 10, and 20 ml. volumes of the stock emulsion. The results are given in Fig. 4.

From previous discussion it may be expected that the rate of exhaustion of oil will be proportional to some function of the initial concentration. This is probably true at low concentrations, but at higher concentrations, the pick-up is limited by the amount of oil (or emulsifying agent) that the wool is capable of adsorbing. It has already been pointed out that the stock emulsion contains a considerable excess of emulsifying agent that is present as free molecules dissolved in the aqueous phase. As the concentration of oil is increased, the concentration of emulsifying agent is also increased, and hence

the amount of oil picked up by the wool does not increase in proportion to the initial concentration of oil. This is shown in Fig. 4, where it is seen that the percentage reduction in emulsion concentration decreases with increasing initial concentration.

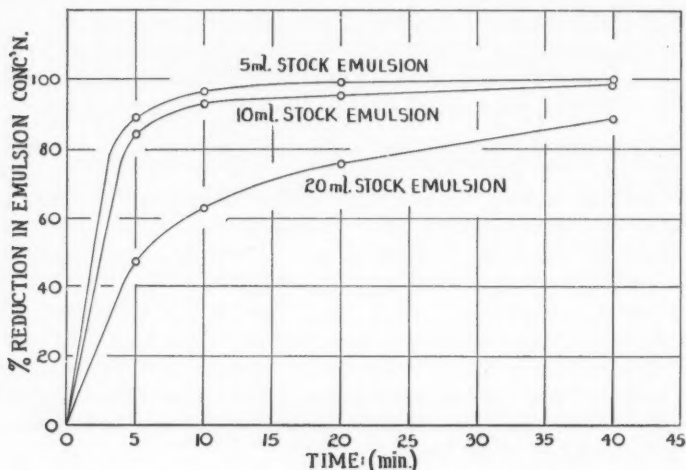


FIG. 4. Influence of concentration of oil on the rate of emulsion exhaustion.

Influence of Soap

Preliminary experiments had shown that small amounts of soap remaining in the wool after laundering and repeated rinsing have a marked effect on the pick-up of oil. The pick-up was greatly improved when this residual soap was destroyed by the inclusion of a souring operation at pH 5.0 prior to the oiling.

In order to study the effect of soap on the oiling process use was made of a launderometer (1). Samples of wool weighing approximately 10 gm. were cut into pieces 2 in. square and laundered at 100° F. (38° C.) according to the following formula.

Operation	Materials	Time, min.
1. Suds	100 ml. of 0.3% neutral soap solution	10
2. Suds	100 ml. of 0.3% neutral soap solution	10
3. Rinse	100 ml. of distilled water	5
4. Rinse	100 ml. of distilled water	5
5. Rinse	100 ml. of distilled water	5

The jars were drained after each operation by inverting over a copper screen. In those cases in which the effect of souring was to be studied, 10 ml. of 0.75% aqueous sodium silicofluoride solution was added to the final rinse

water (Operation 5). After laundering, 100 ml. of distilled water at 100° F. and 3 ml. of the stock emulsion were added to each jar and the jars were returned to the launderometer for a further period of 10 or 60 min. In those cases in which no laundering treatment was given, 100 ml. of water and 3 ml. of stock emulsion were added directly to the dry wool.

After oiling, the wool was removed, allowed to drain into the jar, and then transferred to a weighing bottle. The volume of the residual emulsion was measured, and the sign of the charge on the droplets was determined. This was done by placing some of the emulsion in a small U-tube fitted with two platinum electrodes connected to a 110 volt d-c. line, and observing the direction of movement of the particles. At the conclusion of this test, the contents of the U-tube was returned to the remainder of the residual emulsion, the tube was rinsed out with isopropyl alcohol and petroleum ether, and the total oil content was determined. The wool, after weighing, was air dried and extracted for three hours in a Soxhlet apparatus with petroleum ether. The extracted oil was recovered and weighed. When the first of these tests had been completed, it was observed that the value obtained for the oil content of the wool was in some cases greater than the total weight of oil added in the stock emulsion. This may be explained by the fact that, on the addition of sour, any soap present in the wool or the solution is hydrolysed with the liberation of free fatty acid, which is deposited on the wool. In determining the oil content, this free fatty acid is extracted along with the oil, and hence high results are obtained. In order to correct for this error, the extracted oil plus fatty acid, after weighing, was dissolved in hot, neutral ethyl alcohol and the weight of fatty acid, calculated as stearic acid, was estimated by titration with *N*/10 sodium hydroxide solution. This weight was subtracted from the total weight of extracted material, giving the true weight of oil held by the wool.

In calculating the oil distribution, a distinction was made between oil held by the wool due to preferential adsorption and oil found in the wool merely by virtue of the volume of residual emulsion retained by the wool. In the practical application of the process the bulk of this volume would be removed from the wool by hydro-extraction and it was, therefore, included with the oil found in the residual bath.

The results are given in Table I. The magnitude of the mechanical loss was taken as a criterion of the probable accuracy of the results. When the loss is small, i.e., where nearly all of the oil has been accounted for, it is assumed that the results are of reasonable accuracy.

When the souring operation is omitted, a considerable proportion of the oil is not picked up by the wool and the oil droplets of this residual emulsion are negatively charged. When the souring operation is included, or when the wool is not given a preliminary laundering, the oil pick-up is much greater and the droplets of the residual emulsion are positively charged. In those cases in which a souring operation was included after laundering with soap, the wool was found to contain an amount of free fatty acid equal to approxi-

TABLE I
INFLUENCE OF SOAP ON EMULSION EXHAUSTION

Run No.	Treatment	pH of final rinse	Time of oiling, min.	Oil distribution, % of oil used			Fatty acid found in wool, gm.	Charge on oil drops
				Wool	Residual emulsion	Loss		
1	Laundered	9.5	10	59.2*	38.0	2.8	—	—
2	Laundered	9.4	10	59.7*	38.4	1.9	—	—
3	Laundered and soured	5.0	10	105.7*	8.4	-13.5	—	+
4	Laundered and soured	5.0	10	88.0	7.1	4.9	0.1040	+
5	Oiled only	—	10	91.1	3.4	5.5	0	+
6	Oiled only	—	10	94.5	4.1	1.4	0	+
7	Laundered	9.9	60	69.5	26.0	4.5	0.0061	—
8	Laundered	9.9	60	66.0	28.0	6.0	0.0084	—
9	Laundered and soured	5.1	60	94.5	2.2	3.4	0.0945	+
10	Laundered and soured	5.0	60	91.3	2.3	6.3	0.0927	+
11	Oiled only	—	60	(88.6)	0.4	11.0	0	Sol'n. clear
12	Oiled only	—	60	93.4	0.4	6.2	0	Sol'n. clear

* Not corrected for fatty acid content.

mately 1% of its weight (Table I). It is concluded that this weight of fatty acid was present in the wool as soap prior to the souring operation. It is thus apparent that the presence of soap in the wool causes a reversal of the charge on the emulsion droplets and prevents the complete exhaustion of the bath.

In order to study further the effect of soap on the emulsion, a series of test-tubes were set up each containing 5 ml. of diluted (1 : 20) stock emulsion and increasing amounts of standard soap solution. The total volume was made up to 15 ml. with distilled water. The stock emulsion contained 2 gm. of

Tube No.	1	2	3	4	5	6	7	8	9	10	11
Emulsion, ml.	5	5	5	5	5	5	5	5	5	5	5
Soap sol'n., ml.	0	0.2	0.4	0.6	0.8	1.0	1.2	1.4	1.6	1.8	2.0
Water, ml.	10.0	9.8	9.6	9.4	9.2	9.0	8.8	8.6	8.4	8.2	8.0
Equiv. soap	0	0.17	0.34	0.50	0.67	0.84	1.00	1.17	1.34	1.51	1.68
Equiv. E											

emulsifying agent per 100 ml., from which it is calculated that each tube contained 3.17×10^{-6} equivalents of E. The soap solution was found by analysis to contain 2.66×10^{-6} equivalents of fatty acid per ml. calculated as sodium stearate.

The tubes were shaken and allowed to stand at room temperature. After two hours' standing there was no apparent difference in any of the tubes, but

after four hours, the contents of tube No. 8 showed some evidence of coagulation. After 18 hr. the contents of all the tubes had separated, and, on shaking, the contents of tubes Nos. 6, 7, and 8 became perfectly clear, whereas the others all redispersed giving emulsions of milky appearance. Each emulsion was examined microscopically using a Zeiss cataphoresis cell to determine the relative size of the drops and the sign of their electrical charge. It was found that tube No. 1 contained very small drops having high velocities towards the cathode (positively charged). As the concentration of soap increased (Tubes 2 to 5) an increasing number of large motionless drops were observed with a corresponding decrease in the number of small positively charged drops. The contents of tubes Nos. 6, 7, and 8, having coagulated completely, were not examined. As the soap concentration increased further (Tubes 9 to 11) an increasing number of small negatively charged drops were observed, with a corresponding decrease in the number of large motionless drops.

From the above observations it is concluded that coagulation of the emulsion occurs progressively as the concentration of soap is increased. When soap is present in an amount equivalent to the amount of emulsifying agent, the emulsion is completely coagulated. As the soap concentration is increased above this amount, the emulsion becomes increasingly more stable, with the particles bearing negative charges. It is further to be noted that coagulation of the emulsion does not occur immediately, but requires several hours at room temperature.

These observations are of considerable importance in the practical application of the process. If the wool contains an amount of soap equivalent to or less than the amount of emulsifying agent added, some coagulation of the emulsion occurs, the amount of coagulation depending on the amount of soap present. This does not greatly influence the pick-up of oil, since most of the precipitated oil is deposited on the wool, although there may be some deposition on the walls of the containing vessel. Once this amount of soap has been exceeded, however, the emulsion becomes more or less stable, with the dispersed droplets bearing a negative charge, and hence further exhaustion of the bath by electrostatic attraction is impossible.

Referring to Table I, it is seen that 3 ml. of stock emulsion, which contains 2% of emulsifying agent, was used. The weight of soap equivalent to this quantity of emulsifying agent is thus

$$3 \times \frac{2}{100} \times \frac{25}{100} \times \frac{306}{395.5} = 0.0116 \text{ gm.}$$

Approximately 0.1 gm. of free fatty acid was found in those samples of wool that had been soured (Table I). It is, therefore, clearly evident that sufficient soap is held by the wool after laundering to bring about a reversal of the charge on the emulsion droplets and thus prevent complete exhaustion of the oil.

In the practical application of the process, it is desirable to add approximately 5% of oil to the wool. The treatment of 100 gm. of wool would require

5 gm. of oil, which would be associated with 0.5 gm. of emulsifying agent or 0.1288 gm. of *E*. The weight of soap necessary for the complete coagulation of this quantity of emulsion is $0.1288 \times \frac{306}{395.5} = 0.1$ gm., i.e., for satisfactory oiling the wool must contain not more than 0.1% of soap. If less than 5% oiling is required, or if the ratio of emulsifying agent to oil is reduced, the quantity of soap that can be tolerated is reduced still further.

Notes on the Practical Application of the Process

For the sake of convenience in shipping and storage, stock emulsions containing as high a percentage of oil as possible should be used. Emulsions containing up to 70% of oil by weight have been prepared and found satisfactory. Excess of emulsifying agent should be avoided, both from the standpoint of economy and because of the retarding effect on the rate of exhaustion. Emulsions containing 2.5% of emulsifying agent based on the weight of the oil have been found satisfactory, and it is probable that stable emulsions could be prepared containing a somewhat lower percentage of emulsifying agent.

While higher temperatures result in an increased rate of exhaustion, the temperature that can be used in practice is limited to 100° to 110° F. owing to danger of shrinkage and felting of the wool, if subjected to mechanical action at elevated temperatures. The adverse effect of soap remaining in the wool after laundering has already been discussed, and the necessity for an efficient souring operation has been emphasized. While a high pH favours more rapid exhaustion, the pH must be adjusted with caution since there is danger of regenerating soap from the fatty acid deposited on the wool, if alkaline sodium salts are present.

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USE OF RADON FOR INDUSTRIAL RADIOGRAPHY¹

By A. MORRISON²

Abstract

The factors that have kept radon from becoming popular for industrial radiography are discussed and the conditions under which it will be most useful are deduced. Data are given to enable radiographers to find the correct exposure for radon in those instances where it can be best applied.

Introduction

Radon has been suggested frequently as a source of gamma rays for industrial radiography, but has seldom been used in spite of the fact that there are a number of radon (radium emanation) plants scattered around the country from which it could be obtained. In Canada, plants are operated at Montreal, Toronto, Winnipeg, and Saskatoon. Although radon can be compressed into a small spherical bulb, making a source of radiation that more closely approaches the desired point-source than does the ordinary radium capsule of equal initial strength, it has not become popular. The reason for this is partly the inconvenience associated with the use of radon and partly the cost.

Disadvantages of Radon

The inconvenience arises from the decay of radon and the corresponding decrease in the intensity of the gamma rays from the radon source. During a long exposure the decrease is considerable and the exposure time is appreciably longer for radon than for a radium capsule of equal initial strength*. The appropriate exposure for a radon bulb can be found by first finding the exposure required for a radium capsule of the same initial strength from the usual charts or graphs and then, by finding in a table, such as that given below, the exposure time for radon that corresponds to the exposure required for radium. For example, an exposure that requires 40 hr. with radium, requires 47.7 hr. with radon.

Radon is less convenient than radium when a succession of objects of about the same size and thickness or a number of identical objects are to be radiographed. With radium, by the choice of the appropriate source to film distance and the appropriate film, a few techniques can be developed so that short exposures during the day and long exposures overnight can be used. Once these techniques are determined, no further calculations are necessary. With radon the decreasing strength of the source means that no two exposures

¹ Manuscript received June 14, 1945.

Contribution from the Department of Physics and Electrical Engineering, National Research Laboratories, Ottawa, Canada. Issued as N.R.C. No. 1334.

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* The gamma radiation from 1 mgm. of radium is the same as that from 1 mc. of radon.

can be identical. A gradual increase in the time of exposure or a gradual decrease in the source to film distance, or the use, after a time, of a faster film, is required, and no simple system of techniques can be developed. Each exposure is an individual problem and it is difficult to plan a sequence of exposures that can all be started and finished during normal working hours. This is a serious handicap in a foundry in which radiography is in continual rather than occasional use.

After a period of decay the strength of the radon bulb drops past the point at which it is economical to continue to use the bulb, and it must be discarded and a new one obtained. If for any reason radiography is interrupted, the radon continues to decay, and by the time radiography is to be started again the strength of the radon may have dropped below its useful value. It is necessary to plan in advance to have a bulb of adequate strength on hand when radiography is to begin. In using radon, the factor of decay makes necessary more planning of the individual exposures, more planning of successive exposures, and more planning in the case of any interruptions, than is the case with radium.

Radon is not always available when wanted, since the primary purpose of the emanation plants is the supply of radon for cancer treatment, and this need must be met before any radon is available for industrial work. Thus, if a source of 150 mc. strength is needed for a certain day, there is no certainty that such a source will be available, for the emanation plant must first fill whatever orders there are for seeds or plaques, and must keep its own stock of seeds at a reasonable level.

Cost of Radon

The rental rate for radium is about 35 cents per milligram per month. This is sufficiently low that if radium is in use more than just occasionally the charge for radon must be very moderate to make it worth while. For example, a radon bulb, starting at 100 mc. strength will, over a period of about $8\frac{1}{2}$ days, give about the same number of mc-hr. (10,400) as a 50 mgm. radium capsule. If this were all the use made of the radium during the month, the cost would be \$17.50 for the radium and, correspondingly, $17\frac{1}{2}$ cents a millicurie could be paid for the radon. But if greater use were made of the radium or if it were used at spaced intervals, the radon would need to cost less than $17\frac{1}{2}$ cents a millicurie.

To have for continuous radiography a radon source equivalent to 50 mgm. of radium would require the purchase of at least three bulbs of radon a month each with an initial strength of 100 mc. These should cost not more than the monthly rental of a 50 mgm. capsule, i.e., \$17.50 or about 5 cents to 6 cents a millicurie. Even at this low rate, the other disadvantages associated with radon would probably mean that radium, if available, would be preferred.

Advantages of Radon

Radon does have characteristics that make it particularly suitable in some applications. The small size into which radon of a large initial strength can be compressed is particularly important in cases where the defects are small and are at a relatively large distance from the film, thus making necessary sharp definition, i.e., a source approaching a point-source. The small size of the radon bulb is also an advantage where, because of the shape of the object, it must be placed with a very short source to film distance. In radiographing a hollow steel tube of external diameter 8 in. and wall thickness 2 in. a radon bulb of diameter 1 to 2 mm. is to be preferred to a radium source of two to three times this diameter, since the distance from the part of the steel tube nearest to the source of radiation to the film is about half the distance from the source to the film.

The fact that a strong source of radiation, needed for a specific radiographic inspection, can be obtained without delay from a nearby plant, is a considerable advantage, particularly in areas where radium capsules must be obtained from a great distance and only after considerable correspondence. In many cases, radiographic inspection may require only a dozen exposures which might be made over a period of two or three days with radon. These exposures could be completed before arrangements could be made to obtain a radium capsule. Radium capsules are not always immediately available on demand and in some cases a delay of several weeks is involved.

Conclusions

In view of these considerations it would seem that radon should not be expected to compete with radium when there is to be continuous use made of radiography. Radon should be available and should be used when it is not practical to keep radium continuously on hand, or when the cost of keeping on hand a radium source is greater than the cost of occasionally buying a strong radon source.

To take advantage of its most valuable characteristic, the small size of the source of radiation, the technician in charge of the emanation plant must make every effort to compress the maximum quantity of radon into the smallest possible bulb. A 100 mgm. radium source has a diameter of about 4 mm. and a 250 mgm. source has a diameter of about 6 mm. A radon source of diameter of 1 to 2 mm. for 100 mc. would be highly desirable.

Information for Those Who Use Radon

For the convenience of those who may wish to use radon, two tables and a graph are given below. The first table and graph give the exposure required with radon corresponding to that required for radium. The second table gives the decay of radon and the total number of millicurie-hours of radiation obtained.

The second and fifth columns of the second table give the amount of radon present at any time after the amount was 1.000. If the strength of a radon bulb is known at any time, its strength at any later time can be calculated using this table. For example if the strength of a bulb was known to be 107 mc. at a certain time, its strength 19 hr. later would be $107 \times 0.866 = 92.6$ mc. The strength of this bulb 10 hr. later still could be computed from its original strength multiplied by the decay factor for 29 hr., i.e. $107 \times 0.803 = 85.9$ mc. or, alternatively, by multiplying its strength at 19 hr. by the decay factor for 10 hr., i.e., $92.6 \times 0.927 = 85.9$ mc.

Similarly the exposure in millicurie-hours for the first 19 hr. would be 17.70 per initial millicurie, or a total of $107 \times 17.70 = 1893$ mc-hr. A further 10 hr. would add 9.63 mc-hr. per millicurie strength at the beginning of the 10 hr. period, or a total of $92.6 \times 9.63 = 892$ mc-hr. Or, by using the difference between the total millicurie-hours per initial millicurie for 29 hr., and the total millicurie-hours per initial millicurie for 19 hr., the total would be $107 \times (26.04 - 17.70) = 892$ mc.

In calculating an exposure with radon the following steps would be made:

(1) The strength of the radon at the time the exposure is to be started would be found from its strength at any earlier time and from the appropriate decay factor from Table II.

(2) The exposure time that would be required if a radium capsule of the same strength were used would be found from the usual exposure graphs, in hours.

(3) The corresponding time for radon would be found from Table I, or from the graph.

An alternative method would be as follows:

(1) The strength of the radon source at the time the exposure is to be started would be found from its strength at any earlier time and from the appropriate decay factor from Table II.

(2) The exposure time that would be required if a radium capsule of the same strength were used would be found from the usual exposure graphs, in milligram-hours, i.e., in millicurie-hours.

(3) The number of millicurie-hours of exposure required per initial millicurie would be found by dividing the result of (1) above by the result of (2) above.

(4) From Table II the length of exposure required to get this number of millicurie hours of radiation per initial millicurie would be found.

For example, a radon bulb of 92 mc. strength at 10.00 a.m. is to be used at 5.00 p.m. to radiograph 3 in. of steel at a source to film distance of 15 in. using a medium speed screen-type film (Kodak "Bluebrand"), and a film density of 1.5 is desired. The exposure time required is worked out below.

First Method

- (1) Strength of radon at 5.00 p.m. = $92 \times 0.949 = 87$ mc.
- (2) Exposure time for 87 mgm. of radium = 12.9 hr. (1,2).
- (3) Corresponding time for radon = 13.6 hr. from Table I.

Second Method

- (1) Strength of source = $92 \times 0.949 = 87$.
- (2) Exposure in milligram-hours = 1125.
- (3) Millicurie-hours per initial millicurie = 12.9.
- (4) Exposure time required with radon = 13.6 hr.

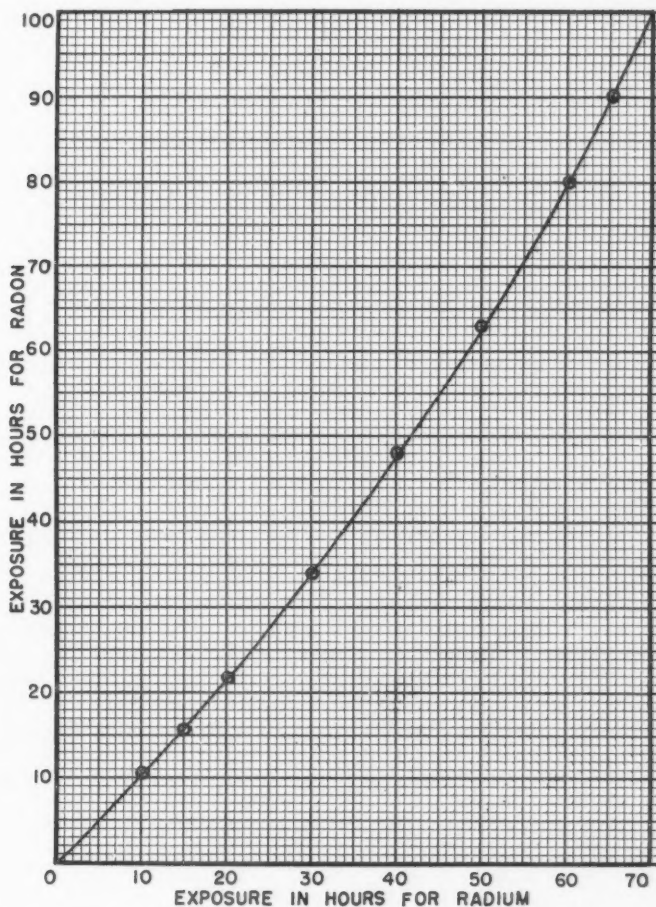


FIG. 1. Graph showing the relation between exposure time required when using radium and that required when using an equal initial strength of radon.

TABLE I

EXPOSURE REQUIRED WITH RADON CORRESPONDING TO VARIOUS EXPOSURES
REQUIRED WITH RADIUM

Exposure with radium	Exposure with radon	Exposure with radium	Exposure with radon
1.0	1.0	15.0	15.9
2.0	2.0	16.0	17.1
3.0	3.0	17.0	18.2
4.0	4.1	18.0	19.4
5.0	5.1	19.0	20.5
6.0	6.1	20.0	21.7
7.0	7.2	22.0	24.1
8.0	8.3	24.0	26.5
9.0	9.3	26.0	28.9
10.0	10.4	28.0	31.5
11.0	11.5	30.0	34.0
12.0	12.6	32.0	36.6
13.0	13.7	34.0	39.3
14.0	14.8	36.0	42.0

TABLE II

DECAY OF RADIUM EMANATION AND CUMULATIVE EFFECT (FOR $\lambda = 0.1812$
PER DAY OR $= 0.00755$ PER HR.)Values of $e^{-\lambda t}$ from tables in *Radioaktivität* by Meyer and Schweidler

Time			$e^{-\lambda t}$	Summation	Time			$e^{-\lambda t}$	Summation
Day	Hour	Hours			Day	Hour	Hours		
0.0	0	0	1.000		1.0	6	30	.797	26.84
	1	1	.993	.996		7	31	.791	27.64
	2	2	.985	1.985		8	32	.785	28.43
	3	3	.978	2.966		9	33	.779	29.21
	4	4	.970	3.940		10	34	.773	29.98
	5	5	.963	4.907	1.5	11	35	.768	30.75
	6	6	.956	5.866		12	36	.762	31.02
	7	7	.949	6.818		13	37	.756	32.28
	8	8	.941	7.763		14	38	.750	33.03
	9	9	.934	8.702		15	39	.745	33.78
	10	10	.927	9.632		16	40	.739	34.52
0.5	11	11	.920	10.56	2.0	17	41	.734	35.26
	12	12	.913	11.47		18	42	.728	35.99
	13	13	.906	12.38		19	43	.723	36.71
	14	14	.900	13.29		20	44	.717	37.43
	15	15	.893	14.18		21	45	.712	38.15
	16	16	.886	15.07	2.0	22	46	.706	38.86
	17	17	.880	15.95		23	47	.701	39.56
	18	18	.873	16.83		0	48	.696	40.26
	19	19	.866	17.70		2	50	.687	41.64
	20	20	.860	18.56		4	52	.675	43.00
	21	21	.853	19.42		6	54	.665	44.34
	22	22	.847	20.27		8	56	.656	45.66
	23	23	.841	21.11		10	58	.645	46.97
1.0	0	24	.834	21.95		12	60	.636	48.25
	1	25	.828	22.78		14	62	.627	49.51
	2	26	.822	23.61		16	64	.617	50.75
	3	27	.816	24.42		18	66	.608	51.97
	4	28	.809	25.24		20	68	.599	53.18
	5	29	.803	26.04		22	70	.589	54.37

TABLE II—Continued

DECAY OF RADIUM EMANATION AND CUMULATIVE EFFECT (FOR $\lambda = 0.1812$
PER DAY OR = 0.00755 PER HR.)—Continued

Values of $e^{-\lambda t}$ from tables in *Radioaktivität* by Meyer and Schweidler—Continued

Time			$e^{-\lambda t}$	Summation	Time			$e^{-\lambda t}$	Summation
Day	Hour	Hours			Day	Hour	Hours		
3.0	0	72	.587	55.55	10.0	0	240	.163	110.8
	2	74	.573	55.71		6	246	.156	111.8
	4	76	.563	57.84		12	252	.149	112.7
	6	78	.555	58.96		18	258	.143	113.6
	8	80	.548	60.06	11.0	0	264	.136	114.4
	10	82	.538	61.15		6	270	.130	115.2
	12	84	.530	62.22		12	276	.124	116.0
	14	86	.523	63.27		18	282	.119	116.7
	16	88	.514	64.32	12.0	0	288	.114	117.4
	18	90	.507	65.34		12	300	.104	118.7
	20	92	.500	66.35	13.0	0	312	.095	119.9
	22	94	.491	67.34		12	324	.087	121.0
4.0	0	96	.484	68.32	14.0	0	336	.079	122.0
	4	100	.470	70.22		12	348	.072	122.9
	8	104	.456	72.07	15.0	0	360	.066	123.7
	12	108	.442	73.87		12	372	.060	124.5
	16	112	.429	75.61	16.0	0	384	.055	125.2
	20	116	.417	77.31		12	396	.050	125.8
5.0	0	120	.404	78.95	17.0	0	408	.046	126.4
	4	124	.392	80.54		12	420	.042	126.9
	8	128	.380	82.08	18.0	0	432	.038	127.4
	12	132	.369	83.58		12	444	.025	127.8
	16	136	.358	85.04	19.0	0	456	.032	128.2
	20	140	.348	86.45		12	468	.029	128.6
6.0	0	144	.337	87.82	20.0	0	480	.027	128.9
	4	148	.327	89.15		12	492	.024	129.3
	8	152	.317	90.44	21.0	0	504	.022	129.5
	12	156	.308	91.69		12	516	.020	129.8
	16	160	.299	92.90	22.0	0	528	.019	130.0
	20	164	.290	94.08		12	540	.017	130.2
7.0	0	168	.281	95.22	23.0	0	552	.015	130.4
	4	172	.273	96.33		12	564	.014	130.6
	8	176	.265	97.40	24.0	0	576	.013	130.8
	12	180	.257	98.45		0	600	.011	131.1
	16	184	.249	99.46	25.0	0	624	.009	131.3
	20	188	.242	100.4		0	648	.007	131.5
8.0	0	192	.235	101.4	26.0	0	672	.006	131.7
	6	198	.224	102.8		0	696	.005	131.8
	12	204	.214	104.1	27.0	0	720	.004	131.9
	18	210	.205	105.3		0	840	.002	132.3
	0	216	.196	106.5	30.0	0	960	.0007	132.4
	6	222	.187	107.8		0			
9.0	12	228	.179	108.8					
	18	234	.171	109.8					

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